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BITUMINOUS MACADAM SURFACING ON A SECTION OF US 60 IN WEST VIRGINIA

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#### Abstract

The reports of research published in this magazine are necessarily qualified by the conditions of the tests from which the data are obtained. Whenever it is deemed possible to do so, generalizations are drawn from the results of the tests; and, unless this is done, the conclusions formulated must be considered as specifically pertinent only to described conditions.


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#### Abstract

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# A STUDY OF ROAD TARS 

## BY THE DIVISION OF TESTS, BUREAU OF PUBLIC ROADS

Reported by R. H. LEWIS, Associate Chemist, and J. Y. WELBORN, Junior Highway Engineer

THE Federal specifications used by the various agencies of the Federal Government include a number of standard specifications for tar products used in road construction. The road tar specifications that are in current use are summarized in table 1. This table lists the specification designation, the requirements for the physical and chemical properties of materials meeting particular grades, and the kinds of tars to be used in the preparation of the finished products.

While there may be considerable variation in test limits, the tests required by the Federal specifications are typical of those usually included in tar specifications throughout the United States. Some English and other European specifications limit the amount of ammonia, tar acids, naphthalene, and anthracene in their road tars, but American specifications generally do not.

The characteristics of the various grades of tars are dependent on the physical and chemical properties of the base tars used in their preparation. As shown in table 1, the materials that are generally specified for production of road tars are coal tars, either gas-house or coke-oven, and water-gas tars. The standard definitions of these products, A. S. T. M. designation D 8-33 are as follows:

Gas-house coal tar: Coal tar produced in gas-house retorts in the manufacture of illuminating gas from bituminous coal.

Coke-oven tar: Coal tar produced in byproduct coke ovens in the manufacture of coke from bituminous coal.

Water-gas tars: Tars produced by cracking oil vapors at high temperatures in the manufacture of carbureted water gas.

The properties of these tars can be greatly altered not only by the character of the oil or coal used but also by the methods and temperatures involved in the various processes used in their production. For instance, for many years carbureted water gas was manufactured
using gas oil, a fraction of petroleum distillate coming off immediately after the kerosene fraction. In recent years fuel oil, which is either a heavier distillate or a mixture of distillate with a base petroleum, has also been utilized for carburetion. The tars resulting from the use of the two types of oil have widely different properties.
J. Edwards has stated ${ }^{1}$ that while the usual laboratory tests are of value, future control must not be limited to laboratory tests only. He intimated that the amount and kind of base tar and the kind and quantity of blending oils should also be specified, since these are vital factors that determine the suitability of the tar for a particular use. Some States have already adopted such specifications. The following 1934 specification of a State highway department for a coal-tar cutback is of this type and not only sets specification limits for the finished product but also specifies the test characteristics of the base and flux used in producing the material.

## state specification for coal-tar cutback discussed

This specification covers coal-tar cutback for treatment of earth roads as follows: This material shall contain not less than 40 nor more than 90 percent by volume of refined coal-tar base, fluxed with a tar material (liquid at $60^{\circ} \mathrm{F}$.), which shall make a homogeneous mixture. The flux shall be a water-gas tar or either distillates of water-gas tar or coal tar or a combination of water-gas tar and the above distillates. The flux, base, and mixture shall conform to the requirements shown in table 2, in which the specific viscosity of the mixture will be subject to variation within the limits designated, as may be directed.

[^0]Table 1.-Summary of Federal specifications for tar products used in road construction

| Federal specification designation | Consistency |  |  |  | $\begin{gathered} \text { Specific } \\ \text { gravity } \\ 25^{\circ} / 25^{\circ} \mathrm{C} \text {. } \end{gathered}$ |  | Total distillate (by weight)- |  |  |  | Softening point of distillation residue | Water | Produced from |
| :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: |
|  | Engler specific viscosity |  | Float test |  |  |  |  |  |  |  |  |  |  |
|  | $\begin{gathered} \text { At } 40^{\circ} \\ \text { C. } \end{gathered}$ | $\begin{gathered} \text { At } 50^{\circ} \\ \text { C. } \end{gathered}$ | $\begin{gathered} \text { At } 32^{\circ} \\ \mathbf{C} \end{gathered}$ | $\begin{gathered} \text { At } 50^{\circ} \\ \text { C. } \end{gathered}$ |  |  |  |  |  |  |  |  |  |
| TC-1-25 | $\begin{array}{r} 8-13 \\ 13-18 \\ 18-25 \\ 25-35 \end{array}$ | $\begin{aligned} & 16-26 \\ & 26-36 \end{aligned}$ | Seconds | Seconds | $1.090+$$1.090+$$1.100+$$1.10-1.18$$1.10-1.22$$1.10-1.22$ | $\begin{array}{r} \text { Percent } \\ 90+ \\ 90+ \\ 90+ \\ 90+ \\ 87+ \\ 87+ \end{array}$ | $\begin{array}{r}\text { Percent } \\ 7- \\ 5= \\ 5- \\ 3- \\ 5- \\ 5- \\ \hline-\end{array}$ | Percent | $\left\lvert\, \begin{array}{r} \text { Percent } \\ 32- \\ 300 \\ 30- \\ 30- \\ 25- \\ 25- \end{array}\right.$ | $\left\lvert\, \begin{array}{r} \text { Percent } \\ 42- \\ 40- \\ 40- \\ 40- \\ 335- \\ 235- \end{array}\right.$ | $\begin{array}{r} \circ{ }^{\circ} \text { C. } \\ 60- \\ 60- \\ 60- \\ 60- \\ 35-60 \\ 35-60 \end{array}$ | Percent $2-$$2-$$2-$$2-$$2-$$2-$ |  |
| TC-2-25 |  |  |  |  |  |  |  |  |  |  |  |  | Gas-house, coke-oven, water-gas and/or |
| TC-4-2 |  |  |  |  |  |  |  |  |  |  |  |  |  |
| TM-1. |  |  |  |  |  |  |  |  |  |  |  |  | Gas-house, coke-oven, and/or water-gas |
| TM-2 |  |  |  |  |  |  |  |  |  |  |  |  | tars. ${ }^{1}$, |
| TR-1-25 | 35-60 |  |  |  | 1. 09-1. 19 | $80+$ | 2-8 | $8-20$ | 16-28 | $36-$ | $65-$ |  | $\left\{\begin{array}{c}\text { Refined gas-house, coke-oven and/or } \\ \text { water-gas tars fluxed with suitable }\end{array}\right.$ |
| TR | 60-80 |  |  |  | 1-10-1. 20 | $80+$ | 2-8 | 8-20 | 16-28 | 36- | $65-$ | $2-$ | water-gas distillates. ${ }^{1}$ |
| TH-1 ${ }^{3}$ |  |  | 60-150 |  | $1.140+$ | $85+$ | 1- |  | 15- | $25-$ | 65- | 0 | Gas-house, coke-oven,water-gas,and/or similar tars. ${ }^{4}$ |
| TP-1-25 |  |  |  | 100-160 | 1.15-1. 20 |  | 1- |  | $10-$ | $20-$ | $65-$ |  |  |
| TP-2-25 |  |  |  | 100-160 | 1. $20-1.26$ | 80-95 | $1-$ |  | 10- | $20-$ | $65-$ | 0 | Gas-house and/or coke-oven tar. ${ }^{4}$ |
| TP-3-25 |  |  |  | 130-190 | 1. 15-1.20 | ${ }^{95}$ 95+ | 1- |  | $10-$ | $20-$ | $65-$ | 0 | Refined water-gas tar. 4 |
| TP-4-25 |  |  |  | 130-190 | 1. 20-1.26 | 80-95 | 1- |  | 10- | $20-$ | $65-$ | 0 | Gas-house and/or coke-oven tar. 4 |
| TP-5-25 |  |  |  | $160-220$ | 1. $15-1.20$ | ${ }_{80-95}^{95}+$ | $1-$ |  | ${ }_{10-}^{10}$ | $20-$ $20-$ | 65- | 0 | Refined water-gas tar.4 Gas-house and/or coke-oven tar. 4 |
| TP-6-25.. |  |  |  | 160-220 | 1. $20-1.26$ | 80-95 |  |  |  | 20 | $65-$ | 0 | Gas-house and/or coke-oven tar.4 |

[^1]75183-36--1

Table 2.- Requirements for flux, base, and mixture of a coal-tar cutback

|  | Flux |  | Base |  | Mixture |  |
| :---: | :---: | :---: | :---: | :---: | :---: | :---: |
|  | Mini- mum | Maximum | Minimum | Maximum | Minimum | Maximum |
| Water, percent by weight. |  |  |  |  |  | 2.0 |
| Specific gravity at $25^{\circ} / 25^{\circ} \mathrm{C}$.... | 1.00 | 1. 12 | 1. 14 | 1. 22 | 1.11 | 1. 16 |
| Specific viscosity, Engler: <br> 50 cubic centimeters at $50^{\circ} \mathrm{C}$ |  |  | 6.0 | 30.0 |  |  |
| 50 cubic centimeters at $40^{\circ} \mathrm{C}$ - | 1.1 | 3.6 |  |  | 5.0 | 8.0 |
| Soluble in carbon disulphide, percent by weight | 95 |  | 88 | 97 | 89 | 98 |
| Distillation (A. S. T. M.): $0-170^{\circ} \mathrm{C}$., per cent by weight. $0-300^{\circ} \mathrm{C}$., per cent by weight. | 25 | 7 87 |  | $35^{4.5}$ |  | 7 45 |
| Specific gravity at $38^{\circ} / 38^{\circ} \mathrm{C}$. of total distillate (water free) to $300^{\circ} \mathrm{C}$ |  |  | 1.00 |  | 0.96 |  |
| Softening point of distillation residue, ${ }^{\circ}$ C. (ring and ball method) |  |  |  | 65 | 35 | 60 |

Where the consumer has adqeuate facilities for plant inspection, a specification of this type may be advantageous. However, with the usual laboratory control, test requirements on the finished products will usually prove satisfactory. As shown in table 1, the Federal specifications give the tar refiners considerable leeway in the manufacture of the various grades of road tar from their available sources of crude tar. With the exception of the TP grades 1,3 , and 5 , that require the materials to be refined water-gas tar, and the TP grades 2,4 , and 6 , that require the materials to be refined from suitable gas-house and/or coke-oven tars, all other grades allow the producer to use a tar from a single source or to blend such materials as any of the above with or without suitable distillates as may be necessary to meet the specification.

The Federal specifications, as well as specifications of the special type already described, control the quality and grade of tar products suitable for various types of road construction by consistency tests, such as the viscosity and float tests, by determination of free carbon, ${ }^{2}$ and by an accelerated laboratory evaporation test. The evaporation test in general use for all tar products is the distillation test, which not only gives the amount of total volatile matter present in the tar but also indicates the probable rate at which these volatile constituents may be lost under service conditions. A softening point determination on the residue from distillation has been considered as a measure of consistency of the material that would be present as active binder when the volatile matter has evaporated from the road surface.

In order to determine how present-day tars conform to the Federal specifications, 35 tar products were obtained from 5 of the leading tar refiners. The laboratory study of these materials is the subject of this report.

## TYPICAL GRADES OF ROAD TAR FROM MAJOR PRODUCERS STUDIED

In table 3 the samples of these road tars are grouped according to their conformity to Federal specification grades, and the composition of the materials as reported by the producers is shown. The intended use of the Federal specification grades, as well as the use recommended by the producers, are also given. Samples 22, 23 , and 31 , submitted by producer C and designated as "special", did not meet any particular specification.
${ }^{2}$ Free carbon in tars is defined by the American Society for Testing Materials designation D 8-33 as organic matter that is insoluble in carbon disulphide. The
term will be so used in this report.

Table 3.-Comparison of road tars with standard Federal specifications


[^2]One or more of the other samples represented each of the specification grades shown in table 1, except grades TP-3 and TP-5. These grades are for refined watergas tars that have the same range in consistency as the coal tar grades TP-4 and TP-6, respectively.

The laboratory study of these road tars included tests to determine their conformity with the particular Federal specifications, and other tests which, although not usually made on road tars, might develop information of value relative to their physical and chemical characteristics. In order to determine how these materials lose their volatile matter under accelerated weathering conditions and to determine the character of the resulting residues, all of the tars were exposed in thin films to the action of air, light, and solar heat. The residues from exposure and the distillation residues were subjected to the same laboratory tests so that the physical and chemical characteristics of the residues obtained by both methods might be compared.

In order to determine the binding values of the original materials and of their distillation residues, Hub-bard-Field stability cylinders were made with these materials combined with a standard sand and the stability of these cylinders was determined. Cylinders containing the original tars and exposed under the same conditions as the thin films were tested for stability to determine the development of binding value under accelerated weathering.

The laboratory tests made on the 35 tar products to show their conformity to the various Federal specification grades were as follows:

Specific gravity at $25^{\circ} / 25^{\circ} \mathrm{C}$.
Specific viscosity. Engler, at $40^{\circ} \mathrm{C}$. and $50^{\circ} \mathrm{C}$.
Float test at $32^{\circ} \mathrm{C}$. and $50^{\circ} \mathrm{C}$.
Solubility in carbon disulphide.
Distillation test, A. S. T. M. D 20-30.
Softening point of distillation residue.
The laboratory tests made to obtain data of special interest were:
Specific viscosity, Engler, at $60^{\circ} \mathrm{C}$.
Saybolt-Furol viscosity at $25^{\circ}, 40^{\circ}, 50^{\circ}$, and $60^{\circ} \mathrm{C}$.
Specific gravity at $38^{\circ} / 38^{\circ} \mathrm{C}$. of total distillate.
A. S. T. M. method D $402-34$ T of pouring distillation residue. Softening point of residue obtained under above method.
Ductility at $10^{\circ}, 15^{\circ}, 20^{\circ}$, and $25^{\circ} \mathrm{C}$. on residues from A. S. T. M. D 20-30' distillation.

The A.S. T. M. and A. A. S. H. O. designations of methods of making these tests are given in table 4.

Table 4.-Tests methods used in studying road tars

| Test | Method of test |  |
| :---: | :---: | :---: |
|  | A. S. T. M. designation | A. A. S. H. O. designation |
| Specific gravity | D 70-27.- | T-43. |
| Specific viscosity, Engler |  | T-54. |
| Saybolt-Furol viscosity | D 88-33_.- | T-72. |
| Float test.. | D 139-27-- | T-50. |
| Poftening point, ring and ball | D $56-25 \ldots \ldots$ | T-49. |
| Ductility | D 113-26T. |  |
| Solubility in $\mathrm{CS}_{2}$ | D 4-27.... | T-44. |
| Water--- | D 95-30_.- | T-55. |
| Distillation |  | $\mathrm{T}-52$ |
| Do | D 402-34 T. | T-78. |

TARS STUDIED WERE IN GENERAL CONFORMANCE WITH FEDERAL SPECIFICATIONS
The results of laboratory tests made to determine conformity with Federal specifications are given in table 5 and show that only four of the road tars failed to meet all of the requirements of the specifications for
the appropriate grades of material. The softening point of the distillation residue of sample 17 was $0.4^{\circ} \mathrm{C}$. over the maximum limit for TC-4 material. The amount of distillate of sample 27 up to $170^{\circ} \mathrm{C}$. was 0.51 percent under the minimum limit for TR-2 material. The float of sample 28 was 215 seconds, or 65 seconds greater than the specification requirement for TH-1 material. Sample 13 failed to meet the specification for TP-1 material which requires the solubility in carbon disulphide to be greater than 95 percent. The three specifications for TP materials produced from water-gas tars require high solubility in carbon disulphide.
Samples 22, 23, and 31 did not meet any particular specification. The tests on sample 22, a heavy, dehydrated coal tar, indicated that it had too high a free carbon content to be satisfactorily processed, without fluxing, to meet any of the Federal specifications. Sample 23 was a very thin, water-gas tar, too fluid for direct use in road construction. Sample 31, while meeting the specification for TH-1 material except that its residue from distillation had too high a softening point, was submitted with sample 23 as the base and flux, respectively, which producer C used in the road tars represented by samples 14 to 19 inclusive. Using the specific gravities of samples 23 and 31 and assuming no change in volume when mixing these two products, samples $14,15,16,17,18$, and 19 contained approximately, by weight, $30,35,40,45,50$, and 55 percent, respectively, of sample 31 as the base in the blended tars. Although the percentage composition of the other blended tars could not be definitely established, the more fluid materials having the lowest densities were perhaps chiefly water-gas tars. The blends of water-gas tars, bunker C water-gas tar, and the blends of coal tar and water-gas tars had high densities.

The percentage of free carbon has been limited to a great extent by specifications, there being still a considerable difference of opinion as to whether a high or a low free-carbon tar is more suitable for a particular purpose. For the heavier grades of tar, such as refined tars of the TP grades, the Federal specifications provide for a coal tar of high free-carbon content and a watergas tar of low free-carbon content. For the other grades, the minimum amount of free carbon permissible is not limited. A study of the solubility of these tars submitted by the various producers indicates that in general the percentage of insoluble material is well under the maximum allowable by the Federal specifications.

Comparing the materials meeting the various specification grades as a group, it will be seen that the TC materials were most soluble in carbon disulphide and the TR and TP materials the least soluble. The TM materials had a solubility close to the TC tars, and the solubility of the TH products was slightly more than that of the TR and TP samples. The average solubilities for the various grades were as follows: TC, 93.56 percent; TM, 92.21 percent; TR, 88.01 percent; TH, 89.58 percent; and TP, excluding sample 13, 88.02 percent. All of the materials, except sample 13, had solubilities in carbon disulphide well within the specification limits for their particular grades.

## MOST FLUID TARS HAD GREATEST LOSS ON DISTILLATION

The distillation test has been generally used in the testing of tars to determine the amount of volatile matter and to determine the probable rate of volatilization of the constituents having low boiling points.

Table 5.-Results of laboratory tests on road tars, showing conformity with Federal specifications

: Federal specification.


Figure 1.-Relations Between Amount of Distillate by Weight and Distilling Temperature for Various Grades of Road Tars.

The average distillation curves for the various grades of road tars are shown in figure 1. For the different materials of the same grade the amount of distillate at the temperatures used for fractionating was fairly close to the average value shown by the curves, except for the TH materials.

It will be seen from figure 1 that there is a relation between the distillation loss at $300^{\circ} \mathrm{C}$. and the consistency of the original material-the TC-1 grade, which was the most fluid, had the greatest loss and the TP-6 grade, which was the most viscous, had the smallest loss. Of the tars for cold application ${ }^{3}$ only the TR-1 and TR-2 grades had a uniform rate of loss from $170^{\circ} \mathrm{C}$. to $300^{\circ} \mathrm{C}$. and both types of material, used primarily for patch work, had distilled off a greater percentage of their total distillate at $170^{\circ} \mathrm{C}$. than did the other cold-application materials. The TC grades had given off a relatively small amount of their total distillate at $170^{\circ} \mathrm{C}$. The curves for TM-1 and TM-2 products are similar to the TC curves.

All of the tars for hot application showed no distillate to $170^{\circ} \mathrm{C}$. and the rate of loss increased from $235^{\circ} \mathrm{C}$.

[^3]to $300^{\circ} \mathrm{C}$. for all grades. The curves, as a whole, show that the cold-application tars contained a relatively high percentage of constituents having low boiling points, and that in service their ultimate consistency is reached almost entirely through the loss of their volatile matter. For the hot-application tars the high boiling point of the contained distillates and the smaller percentage of total distillate to $300^{\circ} \mathrm{C}$., together with the more viscous consistency of the various grades, indicate that their binding properties while increasing with service are, initially, not dependent upon the loss of the more volatile constituents.

Although the Federal specifications for road tars do not contain requirements for the specific gravity of the total distillate except for the TM grades, this requirement appears in many State specifications for tar products. The determination of specific gravity at $38^{\circ} / 38^{\circ} \mathrm{C}$. was made on the recovered distillate of all the tars tested.

The data obtained are tabulated in table 6 and may be of some assistance in identifying the types of materials used in the manufacture of road tars. The materials classed as water-gas tars, which include the mixtures of water-gas tar with bunker C tar, had distillates ranging in specific gravity from 0.948 to 1.004 with an average of 0.982 . The mixtures of coal tar and water-gas tar had distillates ranging in specific gravity from 0.992 to 1.044 with an average of 1.014 . The distillates from coal tars and coal tars thinned with istillate ranged in specific gravity from 0.987 to 1.058 with an average of 1.030 .

There is undoubtedly considerable overlapping in the range in values of specific gravity of the distillates of the tars in each classification. However, as shown in table 6, where two or more types of materials met a single specification grade, the distillates of the watergas tars had the lowest specific gravity, the distillates of the coal tar and water-gas tars, with the exception of the TH-1 grade, were next, and the coal-tar distil-

Table 6.-Specific gravities of total distillates from A. S. T. M. D 20-30 distillation test

lates had the highest. While a specification limit for the specific gravity of the total distillate is only required in the TM grades of the Federal specifications, the test is considered of value by the producers and by others. When properly correlated with other test data it can be used to identify the type of tar. A specific gravity test of the distillate can also be used to detect the presence of petroleum admixtures.

The consistency of the distillation residue is usually determined by a softening-point test. The softening point of the distillation residues ranged from 37.0 to 60.4 , with an average of 49.9 for the cold-application materials, and from 33.1 to 56.1 , with an average of 47.1 for the hot-application materials. It will be seen from table 5 that, in each grade, the road tars from producer E had the softest residues.

SAYBOLT-FUROL VISCOSIMETER SATISFACTORY FOR DETERMINING CONSISTENCIES OF FLUID ROAD TARS
One of the most important characteristics of road tars is the consistency or degree of fluidity. Road tars are used as penetrative treatments, as surface treatments and seals, and as binders in road-mix or premix construction. The ease of application, the workability of the mixes, and the development of stability in the road surface are to a great extent dependent upon the initial consistency of the tar used.

Many different tests to measure the consistency of road tars have been developed. E. O. Rhodes, ${ }^{4}$ working with normal byproduct coke-oven tars, showed the interrelationship of test values obtained with different instruments used to measure consistency. Although he did not stress either the accuracy of the various tests or the speed or ease of making them, he reached the following conclusion: "The use of the Engler specific viscosities at $40^{\circ} \mathrm{C}$. and at $50^{\circ} \mathrm{C}$., and the float tests at $32^{\circ} \mathrm{C}$. and $50^{\circ} \mathrm{C}$., discarding all other consistency measurements, would contribute greatly to the simplification and improvement of road-tar specifications."

The materials meeting the Federal specifications and the road-tar specifications of most States generally have been controlled by a determination of the Engler specific viscosity or the float test as proposed. Although the Saybolt-Furol viscosimeter has been adopted by the State and Federal testing laboratories for determining the consistency of liquid asphaltic road materials, the Engler specific viscosity determination has been generally retained in road-tar specifications. Since the Saybolt-Furol instrument has many advantages over the Engler instrument all of the tars used in this investigation, with the exception of the tars for heavy construction, were tested by both methods. The more viscous products were subjected to the float test. All of the consistency determinations on the original materials are given in table 7.

In order to determine the relation between test values as obtained with the two viscosimeters, the data in table 7 have been plotted in figures 2 and 3 .

In figure 2 the Saybolt-Furol viscosity in seconds at $40^{\circ} \mathrm{C}$., $50^{\circ} \mathrm{C}$., and $60^{\circ} \mathrm{C}$. is compared with the Engler specific viscosity at the same temperature for each particular tar. The points fall quite close to a straight line having a slope of 1 to 4 , that is, the Saybolt-Furol viscosity in seconds at the temperatures used is approximately 4 times the Engler specific viscosity at the same temperature. The viscosity values obtained on
${ }^{4}$ A Discussion of Road Tar Consistency Relationships, Engineering News-Record, vol. 111, no. 16, October 19, 1933.


Figure 2.-Comparison of Saybolt-Furol and Engler Specific Viscosities of Road Tars.

Table 7.-Consistencies of the original materials

| Type of material | Iden-tification | Engler specific viscosity - |  |  | Saybolt-furol viscosity- |  |  |  | Float test - |  |
| :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: |
|  |  | $\begin{gathered} \text { At } \\ 40^{\circ} \mathrm{C} \end{gathered}$ | $\begin{gathered} \mathrm{At} \\ 50^{\circ} \mathrm{C} . \end{gathered}$ | $\begin{gathered} \mathrm{At} \\ 60^{\circ} \mathrm{C} \end{gathered}$ | $\begin{gathered} \mathrm{At} \\ 25^{\circ} \mathrm{C} \end{gathered}$ | $\begin{gathered} \mathbf{A t} \\ 40^{\circ} \mathrm{C} \end{gathered}$ | $\begin{gathered} \mathrm{At} \\ 50^{\circ} \mathrm{C} . \end{gathered}$ | $\begin{gathered} \mathrm{At} \\ 60^{\circ} \mathrm{C} . \end{gathered}$ | $\begin{gathered} \mathrm{At} \\ 32^{\circ} \mathrm{C} . \end{gathered}$ | $\stackrel{\mathrm{At}}{50^{\circ} \mathrm{C} .}$ |
| TC-1.TC-2 | 7 | 9.2 |  |  | Seconds 88 | Seconds | Seconds | Seconds | Seconds | Seconds |
|  | 14 | 10.7 9.5 | 6. 3 |  | 118 | 44 |  |  |  |  |
|  | 24 | 11.8 | 6.3 |  | 167 | 51 | 28 |  |  |  |
|  | 1 | 15.9 | 8. 0 |  | 238 | 65 | 34 |  |  |  |
|  | 15 | 15.5 | 8.5 8.4 |  | 185 | 64 |  |  |  |  |
|  | 33 | 16.2 | 8.0 |  | 342 263 | 73 67 | 36 |  |  |  |
| $\mathrm{T}^{\mathrm{T}} \mathrm{C}-3$ | 16 | 21.2 | 11.2 | 6.6 | 277 | 86 | 47 | 30 |  |  |
|  | 25 | 20.7 | 9.5 |  | 322 | 82 | 40 |  |  |  |
| TC-4 | 3 | 34.9 | 15. 4 | 8.0 | 652 | 142 | 63 | 35 |  | . |
|  | 17 | 32.5 | 16.5 | 9.5 | 490 | 136 | 68 | 40 |  |  |
| 'TM-1 | ${ }_{11}^{8}$ | 40. 1 | 21.1 | 11.6 | 647 | 176 | 84 | 49 |  |  |
|  | 18 | 53.5 48.5 | 24.8 22.6 | 13.4 |  | 192 | 84 93 | 53 |  |  |
|  | 35 | 57.4 | 22. 2 | 10.0 |  | 233 | 90 | 43 |  |  |
|  | 5 | 70.4 | 27.4 | 12.9 |  | 287 | 116 | 54 |  |  |
| TM-2 | 19 | 70.6 | 31.6 | 16.7 |  | 283 | 129 | 68 |  |  |
|  | 26 | 83.7 | 32.0 | 14.5 |  | 334 | 129 | 62 |  |  |
| TR-1 | 9 | 44. 5 | 22.0 | 13.6 |  | 181 | 105 | 55 |  |  |
| T'R-2 | 6 | 60.7 | 26.9 | 12.9 |  | 267 | 114 | 56 |  |  |
|  | 10 | 73. 1 | 32.7 | 18.5 |  | 294 | 149 | 78 |  |  |
|  | 27 | 63.1 | 25.5 | 13.1 |  | 256 | 111 | 56 |  |  |
| TH-1 | ${ }_{12}^{2}$ |  |  | 91.4 |  |  |  | 363 | 129 | 58 |
|  | 12 | ----- | ---- | 110.5 |  |  | ---- | 436 | 102 | 51 |
|  | 32 |  |  | 57.6 |  |  |  | 237 | 80 | 43 |
| TP-1. | 28 |  |  |  |  |  |  |  | 215 | 78 |
| TP-2 | 29 |  |  |  |  |  |  |  |  | 107 |
| $\mathrm{TP}-4$ | 4 |  |  |  |  |  |  | - | 633 | 126 |
| TP-6 | 30 |  |  |  |  |  |  |  | 1,977 | 167 |
| Special | 34 |  |  |  |  |  |  |  |  | 165 |
|  | 22 | 2. 0 |  | 44.7 |  |  |  | 175 | 45 | 30 |
|  | 31 | ----- | -...-- | 142.7 |  |  |  | 520 | 117 | 51 |

some liquid asphaltic materials fall along the same line. This indicates that the relation between the two viscosity tests made on both types of bituminous materials at the same test temperature is quite close.

Frequently it is desired to convert the viscosity at one temperature to the viscosity at another temperature. Many temperature-viscosity conversion tables and charts for both asphaltic and tar products have been published but, as stated by E. O. Rhodes, ${ }^{4}$ they

[^4]

Figure 3.-Relations Between Saybolt-Furol Viscosities of Various Road Tars at Various Temperatures.
are accurate only when materials from the same source and method of manufacture are compared.

The relations existing between the Saybolt-Furol viscosities at different temperatures are shown in figure 3. Because of the varied sources of material and methods of blending no satisfactory average curve could be drawn for all materials, although a definite trend is indicated. This makes it possible to distinguish to a certain degree the source of the various tars. Those materials that are known to be water-gas tars are in
nearly every case above the location of an average curve. Below the average curves are those materials known to be, or interpreted from results of tests to be, coal tars or blends of coal tars. In other words, based on viscosity tests, water-gas tars are somewhat less susceptible to temperature change than are the coal tars or coal-tar blends.

## TEST PROCEDURE FOR TARS SUBSTANTIALLY DIFFERENT FROM THAT FOR ASPHALTIC MATERIALS

The float test at $32^{\circ} \mathrm{C}$. has been generally adopted as a measure of consistency for the tars for hot surface treatments although the data in table 7 show that the use of Saybolt-Furol viscosity at $60^{\circ} \mathrm{C}$. might also be satisfactory. However, for the heavy construction tars of the TP grades, the standard Saybolt-Furol test would not be suitable. The specifications of the American Society for Testing Materials for materials of this type require a softening point determination as a measure of consistency with the option of using the float test at $50^{\circ}$ C., while the Federal specifications control consistency by means of the float test at $50^{\circ} \mathrm{C}$. only. As indicated by table 7 , the use of $50^{\circ} \mathrm{C}$. as the temperature for the float test is to be preferred to $32^{\circ} \mathrm{C}$. because of the greatly reduced time involved in making the test.

On the basis of the data developed by this investigation, it is believed that the float test at $32^{\circ} \mathrm{C}$. and $50^{\circ} \mathrm{C}$. is a satisfactory test for the consistency of the hot-application tars. Since the Saybolt-Furol viscosimeters with thermostatic control can be operated to give close checks on duplicate tests, require less material for test, and greatly shorten the time of testing, the adoption of Saybolt-Furol viscosity for cold application road tars, as has been done for liquid asphaltic road materials, would be a pronounced advance toward the general simplification and standardization of tests for this type of bituminous material.

In connection with this discussion of the measurement of consistency, mention should be made of a new viscosimeter. ${ }^{5}$ This instrument is designed to measure the viscosity of all grades of bituminous material at a single temperature and the results are expressed in terms of the absolute viscosity, the poise. Much preliminary work must be done before general adoption of this new test can be expected, but its possibilities are such that it merits serious consideration. It presents a possible means of overcoming the present disadvantages of using several methods for measuring consistency and several temperatures for each method.
While there do not appear to be any practical reasons why all bituminous materials should not be tested in the same manner, for years the test procedure for tars has been substantially different from that for asphaltic materials. For tars, the amount of volatile matter has been determined by the distillation test and the materials have not usually been submitted to the volatilization test (loss on heating at $163^{\circ} \mathrm{C}$.) which has been extensively used for liquid asphaltic materials. The volatilization test has been discarded rather generally, especially for the more rapid-hardening, liquid asphaltic products, and a distillation test has been substituted to determine the amount and rate of loss of volatile matter.
The method of making the distillation test for liquid asphaltic materials is described in S.S.T.M. Tentative Method of Test for Separation of Liquid Asphaltic
${ }^{6}$ New Viscosimeter for Bitumens Has Extended Range, by E. O. Rhodes, E. W. Volkmann, and C. T. Barker. Engineering News-Record, vol. 115, no. 21, Nov. 21, 1935.

Products, designation D 402-34 T, while the distillation test for tars is A.S.T. M. Standard Method of Test for Distillation of Bituminous Materials Suitable for Road Treatment, designation D 20-30. There are some essential differences that might lead to the belief that the results obtained by the two methods would be radically different.

Both methods use the same flask and thermometer. Method D 20-30 requires an air condenser; method D 402-34 T requires a water condenser. In the tar distillation the top of the bulb of the thermometer is at the bottom of the tubulature and the vapor temperature controls the distillation cuts and end point. In the liquid asphalt distillation the bottom of the bulb of the thermometer is $1 \frac{1}{4}$ inch above the bottom of the flask and the distillation fractions and end point are controlled by the temperature of residue or the liquid temperature. In the distillation of tars a 100 -gram sample is used, while in the distillation of liquid asphaltic materials a 200 -cubic centimeter sample is used.

The percentage of distillate is reported by weight in the tar distillation and by volume in the distillation of the liquid asphaltic material. The end point for the distillation of tar products is $300^{\circ} \mathrm{C}$. ( $572^{\circ} \mathrm{F}$.) vapor temperature. The end point of the distillation of asphaltic materials is $360^{\circ} \mathrm{C}$. ( $680^{\circ} \mathrm{F}$.) liquid temperature. The tar residue is cooled in the flask below $125^{\circ}$ C. $\left(257^{\circ} \mathrm{F}\right.$.) before pouring for further tests. The asphaltic residue is poured as quickly as possible after the end point of the distillation is reached.

In order to make some comparison of the results obtained by the two methods, 100 -gram samples of one tar of each grade were distilled with two thermometers in the flask, so that the vapor temperature at the tubulature and the liquid temperature $1 / 4$ inch above the bottom of the flask could be determined. The relations between the vapor and liquid temperature are shown in figure 4.
METHOD OF POURING DISTILLATION RESIDUES AFFECTS THEIR
The curves indicate quite clearly that for these tars, with one exception, a vapor temperature of $300^{\circ} \mathrm{C}$. $\left(572^{\circ} \mathrm{F}\right.$.) corresponds to a liquid temperature somewhat below $360^{\circ} \mathrm{C}$. $\left(680^{\circ} \mathrm{F}\right.$.). The one exception is the tar of the TC grade for which the vapor temperature of $300^{\circ} \mathrm{C}$. corresponds to a liquid temperature slightly in excess of $360^{\circ} \mathrm{C}$. The rather close agreement between a vapor temperature of $300^{\circ} \mathrm{C}$. and a liquid temperature of $360^{\circ} \mathrm{C}$. indicates that for these tar products there can be no great difference either in the amount of distillate or character of the residue, due solely to the difference between a vapor end point of $300^{\circ}$ ( and a liquid end point of $360^{\circ} \mathrm{C}$.
However, figure 4 does indicate that if arbitrary intermediate cutting points are taken, based on liquid temperature control, the fractions of distillate may be composed of constituents having widely different boiling points. For instance, if a liquid temperature cut is made at $225^{\circ} \mathrm{C} .\left(437^{\circ} \mathrm{F}\right.$.), the fraction below this temperature will contain material with a maximum boiling point in the case of the TC product of $125^{\circ} \mathrm{C}$. ( $257^{\circ} \mathrm{F}$.) and in the case of the TR product of $180^{\circ} \mathrm{C}$. ( $356^{\circ} \mathrm{F}$.), a spread of $55^{\circ} \mathrm{C}$. Also, if a vapor temperature cut is made at $150^{\circ}$ C., the spread between the TR and TP materials is $40^{\circ} \mathrm{C}$. Therefore, it would seem that vapor temperature distillation control would be slightly the more preferable if it is desired to determine the character of the fractions obtained at various cutting temperatures, especially when comparison of


Figure 4.-Comparison of Liquid Temperature One-Fourth Inch Above Bottom of Flask and Vapor Temperature at Tubulature for Various Road Tars.
the distillates from materials of widely different composition is desired.
Similar tests on a few samples of liquid asphaltic materials of the slow-curing, medium-curing, and rapid-curing types gave similar results. The curves showing the relation between liquid and vapor temperatures were of different shape for the different types of materials and also for different materials of the same type. There was also the same general agreement between a vapor temperature of $300^{\circ} \mathrm{C}$. and a liquid temperature of $360^{\circ} \mathrm{C}$., the vapor temperature of $300^{\circ} \mathrm{C}$. corresponding to a liquid temperature of $360^{\circ} \mathrm{C}$. or less.

Having established that there is no wide difference between a vapor end point of $300^{\circ} \mathrm{C} .\left(572^{\circ} \mathrm{F}\right.$.) and a
liquid end point of $360^{\circ} \mathrm{C} .\left(680^{\circ} \mathrm{F}\right.$.), the effect on the character of the residue obtained by pouring as required by A.S. T. M. method D 20-30 and by A. S. T. M. method D 402-34 T was determined. The results of tests made on residues poured after they had cooled below $125^{\circ} \mathrm{C} .\left(257^{\circ} \mathrm{F}\right.$.) and when the residues were poured immediately after a vapor temperature of $300^{\circ}$ C. $\left(572^{\circ} \mathrm{F}\right.$.) was reached, are given in table 8.

The difference in the percentage of volatile matter lost in pouring and cooling the residues by the two methods varied from 0.83 to 4.15 percent of the total sample for all the materials tested, with an average difference of 2.37 percent. The losses in volatile matter in A.S. T. M. method D 20-30 are unavoidable losses caused by retention of distillate in the condenser. The effect of the greater loss of volatile matter that occurs when residues are poured according to A. S. T. M. method D 402-34 T is shown by the harder consistency of the corresponding residues. The softening points of the residues poured immediately were from $2.3^{\circ}$ to $21.1^{\circ}$ C., with an average of $6.8^{\circ} \mathrm{C}$., higher than those poured in the usual procedure for tars. The penetration values also show the greater hardness of the residues poured immediately, the decrease in penetration ranging from 0 to 120 with an average of 26.3. Due to this increase in hardness, the softening point values of six of the residues from the TC and TM grades of tar, were increased until they exceeded the maximum allowed by the Federal specifications.

When an asphalt has been blended with a naphtha to produce a liquid asphalt of the rapid-curing type, it has been found that by pouring the residue immediately after the end point is reached the penetration of the residue is in very close agreement with the penetration of the base asphalt used in producing the blend. However, no information on the consistency of the base materials in medium-curing and slow-curing liquid asphaltic products is obtainable, even with this methorl

Table 8.-Effect of method of pouring distillation residues upon their properties

| Type of material | Identi-fication | Total loss in distillation test |  |  | Loss of volatile matter during test |  |  | Softening point |  |  | Penetration nt $25^{\circ} \mathrm{C}$., $100 \mathrm{~g}, 5 \mathrm{sec}$. |  |  |
| :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: |
|  |  | $\begin{aligned} & \text { A. S. T. } \\ & \text { M. } \\ & \text { D } 20-30 \end{aligned}$ | $\begin{gathered} \text { A.S. T. } \\ \text { M. } \\ \text { D } 402-34 \mathrm{~T} \end{gathered}$ | Difference | $\begin{aligned} & \text { A.S. T. } \\ & \text { M. } \\ & \text { D } 20-30 \end{aligned}$ | $\begin{gathered} \text { A.S. T. } \\ \text { M. } \\ \text { D } 402-34 \mathrm{~T} \end{gathered}$ | Difference | $\begin{gathered} \text { A.S. T. } \\ \text { M. } \\ \text { D } 20-30 \end{gathered}$ | $\begin{gathered} \text { A.S. T. } \\ \text { M. } \\ \text { D } 402-34 \mathrm{~T} \end{gathered}$ | Difference | $\begin{gathered} \text { A.S.T. } \\ \text { D } 20-30 \end{gathered}$ | $\begin{gathered} \text { A.S. T. } \\ \text { M. } \\ \text { D } 402-34 \mathrm{~T} \end{gathered}$ | Difference |
| Tr.1TC: 2 |  | Percent | Percent | Percent | Percent | Percent | Percent | ${ }^{\circ} \mathrm{C}$. | ${ }^{\circ} C$. | ${ }^{\circ} \mathrm{C}$. |  |  |  |
|  | 7 | 35.90 | 37. 29 | 1.39 | 0.75 | 1.58 | 0.83 | 45.0 | 54.0 | 9.0 | 38 | 21 | 17 |
|  | 14 | 38. 04 | 38.90 | . 86 | . 62 | 2. 79 | 2. 17 | 54.8 | 61.7 | 6. 9 | 25 | 9 | 16 |
|  | 20 | 32.06 | 35. 64 | 3. 58 | 30 | 3.30 | 3. 00 | 47.4 | 55.4 | 8.0 | 38 | 12 | 26 |
|  | 24 | 34.35 | 37.95 | 3. 60 | 33 | 2. 58 | 2. 25 | 45.2 | 54.4 | 9.2 | 50 | 13 | 37 |
|  | 1 | 33. 19 | 34. 55 | 1. 36 | . 33 | 2. 59 | 2. 26 | 44.6 | 50.1 | 5. 5 | 38 | 30 | 8 |
|  | 15 | 36. 34 | 37. 66 | 1. 32 | . 56 | 2. 44 | 1.88 | 55.6 | 62.7 | 7.1 | 20 | 8 | 12 |
|  | 21 | 29. 25 | 31.75 | 2. 50 | . 34 | 2. 94 | 2. 60 | 50.8 | 57.0 | 6. 2 | 27 | 8 | 19 |
|  | 33 | 29. 92 | 34. 07 | 4. 15 | . 38 | 2.84 | 2.46 | 37.0 | 58.1 | 21.1 | 138 | 36 | 102 |
| 'T' ${ }^{\text {c }}$ 3. | 16 | 35. 50 | 36. 69 | 1. 19 | 1.00 | 2.83 | 1.83 | 57.1 | 63.1 | 6. 0 | 18 | 7 | 11 |
| 1( | 25 | 31.89 | 34. 15 | 2. 26 | . 55 | 2.89 | 2.34 | 46.6 | 52.2 | 5. 6 | 42 | 19 | 23 |
| 'TC-4 | 3 | 29. 10 | 31.82 | 2. 72 | . 85 | 3. 07 | 2. 22 | 45.0 | 54.0 | 9.0 | 53 | 16 | 37 |
| TM-1. | 17 | 34. 78 | 35. 44 | . 66 | . 57 | 2.65 | 2.08 | 60.4 | 64.4 | 4. 0 | 12 | 5 | 7 |
|  | 8 | 26. 05 | 28.80 | 2. 75 | . 07 | 1.96 | 1. 89 | 41.0 | 48.4 | 7.4 | 71 | 39 | 32 |
|  | 11 | 27. 57 | 30. 06 | 2. 49 | . 44 | 2.32 | 1.88 | 42.0 | 49.2 | 7.2 | 74 | 41 | 33 |
|  | 18 | 33. 42 | 34.59 | 1. 17 | 1. 27 | 3. 40 | 2. 13 | 59.4 | 64.1 | 4.7 | 15 | 6 | 9 |
|  | 35 | 25. 31 | 28. 97 | 3. 66 | . 06 | 4.15 | 4.15 | 39.6 | 48.2 | 8. 6 | 97 | 36 | 61 |
|  | 5 | 25.73 | 27. 74 | 2. 01 | . 65 | 2. 56 | 1.91 | 46.1 | 52.5 | 6.4 | 46 | 18 | 28 |
| TM-2. | 19 | 31.88 | 32. 45 | . 56 | . 80 | 2.31 | 1. 51 | 60.0 | 65.8 | 5. 8 | 12 | $\begin{array}{r}5 \\ \hline\end{array}$ | 7 |
|  | 26 | 23. 40 | 26.07 | 2. 67 | . 36 | 3. 54 | 3.18 | 43.2 | 50.7 | \%. 5 | 62 | 25 | 37 |
| TR-1. | 9 | 27. 75 | 29.82 | 2. 07 | . 45 | 1.92 | 1.47 | 60.0 | 65.0 | 5. 0 | 12 | 7 | 5 |
| ' R -2. | 6 10 | 25. 90 25.52 | 28. 33 | 2. 43 | . 71 | 3. 67 | 2. 96 | 53.1 | 60.4 | 7.3 | 20 | 5 | 15 |
|  | 27 | 25. 45 | 27.36 | 1. 91 | . 29 | 2. 48 | 1. 3.19 | 55.8 55.4 | 61.0 59.5 | 5.2 4.1 | 18 | 9 | 9 9 |
| TH-1 | 2 | 19.80 | 21.89 | 2. 09 | . 57 | 3.31 | 2. 74 | 51.7 | 58.1 | 6.4 | 26 | 9 | 17 |
|  | 12 | 15. 54 | 16. 58 | 1. 04 | . 38 | 1. 55 | 1.17 | 41.6 | 44.8 | 3.2 | 89 | 67 | 22 |
|  | 32 | : 11.12 | 15.71 | 4. 59 | . 31 | 3. 68 | 3.37 | 33.1 | 39.7 | 6. 6 | 218 | 98 | 120 |
|  | 28 | 14.37 | 15.82 | 1. 45 | . 48 | 3.12 | 2. 64 | 47.0 | 52.8 | 5.8 | 43 | 21 | 22 |
| TP-1 | 13 | 11. 79 | 13. 10 | 1. 31 | . 19 | 1. 49 | 1. 30 | 49.0 | 54.0 | 5. 0 | 38 | 22 | 16 |
| TP-2 | 29 | 11. 72 | 14.77 | 3. 05 | . 32 | 3. 68 | 3.36 | 49.6 | 56.9 | 6.4 | 30 | 10 | 20 |
| TP-4. | 4 | 13. 20 | 15. 63 | 2. 43 | . 99 | 4. 74 | 3.75 | 56.1 | 60.1 | 4.0 | 12 | 5 | 7 |
| TP-6 | $\left\{\begin{array}{l}30 \\ 34\end{array}\right.$ | 8. 72 | 10.88 | 2. 16 | . 31 | 3. 57 | 3. 26 | 51.8 | 56.8 | 5. 0 | 25 | 12 | 13 |
| Special | 34 | 5. 90 | 9.38 | 3. 48 | . 18 | 3. 42 | 3.24 | 44.1 | 54.4 | 10.3 | 71 | 13 | 58 |
|  | 22 | 19.62 | 21. 50 | 1. 88 | . 00 | 2.31 | 2. 31 | 58.5 | 61.6 | 3.1 | 15 | 6 | 9 |
|  | ) 23 | 47.85 | 49. 23 | 1. 38 | . 48 | 2.80 | 2. 32 | 39.1 | 52.7 | 13. 6 | 84 | 26 | 58 |
|  | ( 31 | 18. 90 | 19.79 | . 89 | . 79 | 2. 58 | 1.70 | 73.3 | 75.6 | 2.3 | 1 | 1 | 0 |

of pouring. Most road tars have tar pitches as bases which contain some rolatile matter to $300^{\circ} \mathrm{C}$. ( $572^{\circ} \mathrm{F}$.) and it is doubtful if the true consistency of the base material can be approximated by either method of pouring the residue.

Liquid asphaltic materials have been classified according to the consistency of the residues from the 1) 402-34 T distillation test as follows: ${ }^{6}$

Group 1.-Liquid residue, float test less than 25 seconds at $122^{\circ} \mathrm{F} .\left(50^{\circ} \mathrm{C}\right.$.), nonhardening or extremely slow hardening.

Group 2.-Float residue, float test more than 25 seconds at $122^{\circ} \mathrm{F} .\left(50^{\circ} \mathrm{C}\right.$.) and penetration of more than 300 at $77^{\circ} \mathrm{F}$. ( $25^{\circ} \mathrm{C}$. ), medium hardening.
Group 3.-Penetration residue, penctration less than 300 at $77^{\circ} \mathrm{F}$. ( $\left.25^{\circ} \mathrm{C}.\right)$, rapid hardening, the speed of hardening being dependent on the amount and volatility of the distillate.
Under the above classification, it will be seen from table 8 that all of the road tars included in this investigation, even if the softer consistencies of the residues obtained by the I) 20-30 method of pouring are considered, are of the rapid-hardening type of bituminous material.

## Distllation residues had Good ductility at normal TEMPERATURES

For some time specifications for many asphaltic materials have included a ductility requirement as a means of control and as an indication of the adaptability of the products to particular uses. Asphaltic cements, and usually the distillation residues and asphaltic residues of liquid asphaltic materials, are required to show a certain ductility at $25^{\circ} \mathrm{C}$. ( $77^{\circ} \mathrm{F}$.) and for some purposes at lower temperatures such as $1.5^{\circ}$ (. $\left(34.7^{\circ} \mathrm{F}\right.$.) or $4^{\circ}\left(3 .\left(39.2^{\circ} \mathrm{F}\right.\right.$.). While the ductility test has been criticised as not being a good measure of the resistance to the stresses to which asphalts in road surfaces are subjected, its use as a laboratory test for these materials is widespread.

If the asphalts used in particular types of road construction must meet a ductility requirement, it is logical that tar products for similar use should also meet a ductility requirement. At the present time the only tars that are tested for ductility are the tar pitches used in roof construction and for water-proofing structures. Few data are available regarding the ductility of road tars. In order to obtain as much information as possible on the physical properties of these tar products, the residues intained in the standard A. S. T. M. 1) 20-30 distillation test were tested for ductility.

The ductility tests were made on each distillation residue at several temperatures in order to obtain some ider of its temperature-ductility curve. The lests were made at $10^{\circ} \mathrm{C} \% 15^{\circ} \mathrm{C}, 20^{\circ}$ (.), and $25^{\circ} \mathrm{C}$. and the results are given in table 9 .
In order to compare the results of the ductility test with the other tests made on the distillation residues, the data in table 9 have been grouped according to the index shown in table 10 . An index of 1 represents the most ductile residue at low temperature and an index of 7 the least ductile.

The ductility index and the other test results on the distillation residues are given in table 11. It is evident that consistency as measured by the softening point or penetration is related to the ductility of these tar pitches. It a given temperature, the harder the

[^5]material the less ductility it possesses. It is indicated that the free carbon content also influences the ductility: In general, as the percentage of free carbon increases the ductility decreases.

Table 9.-Ductilities of the A. S. T. M. D 20-30 distillation test resilues


Table 10.-Ductility index for distilíation residues of tars

| Index | 1)uctility (cm) |  |
| :---: | :---: | :---: |
| 1. | $90+$ at $10^{\circ} \mathrm{C}$. |  |
| 2. | $55-110+$ at $15^{\circ} \mathrm{C}$. | $0-142$ at $10^{\circ}$ ? |
| 3 | $110+$ at $20^{\circ} \mathrm{C}$. | $0-34$ at $15^{\circ}$ ? |
| 4. | $52-110+$ at $25^{\circ} \mathrm{C}$. | $60-81$ at $20^{\circ} \mathrm{C}$. |
| 5. | $110+$ at $25^{\circ} \mathrm{C}$. | 1/4-3/4 at $20^{\circ} \mathrm{C}$. |
| 6 | 43-79 at $25^{\circ} \mathrm{C}$. | $0-112$ at $20^{\circ} \mathrm{C}$. |
| 7 | 0 at $25^{\circ} \mathrm{C}$. |  |

Table 11.-Range in test values of the A. S. T. M. D 20-30 Distillation test residues grouped according to ductility index


EFFECTS OF EXPOSURE ON TAR PRODUCTS HAVE BEEN STUDIED EXTENSIVELY
W. II. Flood ${ }^{7}$ has stated that the great majority of asphalts of the 40-50, 50-60 and 60-70 grades used in road construction have ductility values beyond the limits of the ductility machine. He has shown that materials meeting these penetration grades have ductility values at $60^{\circ} \mathrm{F} .\left(15.5^{\circ} \mathrm{C}\right.$.) averaging from 10.5

[^6]centimeters to 75.4 centimeters, and at $39.2^{\circ} \mathrm{F}$. ( $4^{\circ} \mathrm{C}$.) values averaging from 3.2 centimeters to 4.8 centimeters for the different consistency ranges and sulphur contents. When these figures are compared to the results obtained on the tar residues having a ductility index of 2 and a range in penetration of 38 to 71 , it is seen that tars have a more rapid and abrupt loss of ductility when the test temperature is reduced than do asphalts. This is the result, no doubt, of the greater susceptibility of tars to temperature change, causing a more rapid transition of these materials from a semisolid to a solid state. The tests show that nearly all of the residues have good ductility at $25^{\circ} \mathrm{C} .$, but, in general, this ductility is rapidly lost when the temperature of test is reduced.

In recent years much thought has been given to the changes that take place in bituminous materials under service conditions. The initial bonding strength of a tar product when applied to the road surface is developed in different ways, depending not only on the type of original material but also on the way it is used and the climatic conditions to which it is subjected. All grades of road tar lose a certain amount of volatile matter. This tends to stiffen or harden the material. After the initial bond has been developed, either by evaporation, cooling, or a combination of both, certain hardening takes place because of inherent changes brought about by weathering.

Early investigators have done much work to substantiate these facts. Hubbard and Reeve ${ }^{8}$ published a paper in 1913 regarding the effect of exposure on bitumens. Included in this investigation were three tar products; namely, a refined coal tar, a refined watergas tar, and a refined mixture of coal tar and water-gas tar. The authors made the following conclusion:

It is apparent from these results that the hardening of all bitumens upon exposure is not due to loss by volatilization of the lighter constituents, alone, although in tars such loss is probably responsible to a considerable degree for the hardening of these products. The hardening of petroleum and asphalt products is undoubtedly due to oxidation which proceeds slowly at comparatively low temperatures but increases at higher temperatures. It seems probable that oxidation also plays a part in the hardening of tars although the preceding data does not absolutely prove this fact.

Later Reeve and Lewis ${ }^{9}$ substantiated and amplified the previous data which showed that upon exposure bituminous materials undergo changes caused by factors other than loss of the more volatile constituents. They also showed that the insoluble organic matter in the bitumens they examined increased materially in a closed oven where the chances of oxidation are reduced to a minimum. The authors stated that while oxidation plays a part in the changes that occur, they were led to the conclusion that polymerization and intermolecular reactions induced by heat, and possibly increased by the action of light, are also very largely responsible for such changes, in addition to those accounted for by simple evaporation.

Reeve and Anderton, ${ }^{10}$ while noting that the action of air and sun upon bitumens in their original state is probably quite different than might be expected in actual service, concluded that: (1) Upon exposure tar

[^7]products harden to a much greater extent than could be attributed to the loss of volatile matter alone; (2) changes are accompanied by formation of material insoluble in carbon disulphide; (3) the development of free carbon was greater in water-gas tar than in coal tar; and (4) the comparative consistency of the residues from distillation to a single arbitrary end temperature did not represent the relative behavior of tars in service.

More recent investigations ${ }^{11} 12$ by the Bureau on the effects of exposure on liquid asphaltic materials of the slow-curing, medium-curing, and rapid-curing types have indicated that many materials that have similar characteristics as determined by the usual laboratory tests behave quite differently when exposed in thin films or in mixtures with a standard aggregate.

Exposure tests on the tars were made in boxes of the same type as were used in previous investigations of the Bureau. A sketch of the box is shown in figure 5. The boxes were constructed of $3 / 4$-inch wood, the interior dimensions being 30 by 24 by 3 inches. A hole in the center of the bottom of each box served to admit a stream of clean, dry air to carry away any collected vapors. Outlets for the air were made by cutting slots in the sides of the boxes. These were packed with cotton batting to prevent the entrance of dust. Plate glass, resting on thin strips of felt placed along the edges of the boxes, served as covers.

Three 50 -cubic centimeter samples of each tar were placed in seamless flat tins, $51 / 2$ inches in diameter and $5 / 8$ inch deep. This amount of material gave a uniform film thickness of $1 / 8$ inch. The exposure boxes containing the three sets of exposure samples, one each for 5-, $10-$, and 15 -week periods, are shown in figure 6 .

## ULTIMATE LOSS ON EXPOSURE CLOSELY APPROXIMATED LOSS IN

 A.S.T.M. D $20-30$ DISTILLATION TESTThe samples were placed in the exposure boxes on June 15, 1933. During the following 15 weeks the hourly temperatures within the boxes were recorded daily from $9 \mathrm{a} . \mathrm{m}$. to $4 \mathrm{p} . \mathrm{m}$. The maximum temperature reached during this time was $196^{\circ} \mathrm{E} .\left(91^{\circ} \mathrm{C}\right.$.) occurring on a clear day with a maximum of sunshine. On cloudy days the temperature within the boxes was the same as the outside air temperature. The average maximum daily air temperature was $85^{\circ} \mathrm{F} .\left(30^{\circ} \mathrm{C}\right.$.). As determined from United States Weather Bureau reports for the period of exposure, the samples from the 5 -, 10 -, and 15 -week periods were subjected to 333 , 611 , and 866 hours of sunlight, respectively.

The samples tested at the end of 15 weeks of exposure were weighed at various intervals to determine the loss of volatile matter at all intermediate periods except the 35 -day and 70 -day periods ( 5 and 10 weeks). The losses at these two periods were determined on the samples removed for test at that time, so that the percentage of loss obtained could be correlated accurately with the test values on the residues. This procedure, no doubt, accounts for variations in losses between the 35 - and 70 -day tests that appear to indicate gains in weights rather than continued losses. The percentages of loss at different periods are tabulated in table 12 and the average for each grade of material is shown graphically in figure 7.

[^8]

Figure 5.-Box Used in Exposure Tests.
Table 12.- Rate of loss of volatile matter in thin-film exposure of road tars

| Type of material | Iden-tification | Time of exposure in days |  |  |  |  |  |  |  |
| :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: |
|  |  | 2 | 8 | 15 | 22 | 35 | 50 | 70 | 105 |
| T C-1 |  | Per- | Per- | Per- | Per- | Per- | Per- | Per- | Per. |
|  |  | cent | cent | cent | cent | cent | cent | cent | cent |
|  |  | 23.9 | 31.4 | 32.4 | 32.9 | 33. 2 | 33.5 | 34.4 | 34.0 |
|  | 14 | 24. 1 | 31.5 | 31.9 | 32.7 | 33.1 | 33.2 | 33.7 | 33.6 |
|  | 214 | 22.3 | 29.5 | 31.2 | 32.9 | 33.9 | 33.9 | 33.3 | 34. 5 |
|  | 24 | 22.4 | 30.5 | 32.8 | 34.9 | 35.3 | 36.0 | 35.3 | 36.3 |
| T ${ }^{*}-2$ | 5 | 19. 2 | 26.6 | 28.7 | 30.9 | 31.4 | 31.8 | 31.1 | 32.3 |
|  | 15 | 21.5 | 30. 1 | 30.3 | 31.0 | 30.5 | 31.4 | 30.7 | 31.7 |
|  | 21 | 18.7 | 26. 1 | 27.4 | 29.4 | 29.2 | 30.1 | 29.1 | 30.7 |
|  | 33 | 17.2 | 24.3 | 27.3 | 30.4 | 30.8 | 31.6 | 32.7 | 30.5 |
| TC | 16 | 21.6 | 29.2 | 29.3 | 29.9 | 28.8 | 30.3 | 29.5 | 30.9 |
|  | 25 | 19.2 | 26.9 | 28.6 | 30.6 | 31.6 | 32.4 | 31.3 | 32.7 |
| TC-4 | 3 | 17.11 | 24.1 | 25.9 | 27.4 | 26.2 | 28.5 | 23.4 | 28.6 |
| TM-1 | 17 | 21.6 | 26.8 | 27.0 | 27.5 | 26.0 | 28. 2 | 27.1 | 28.4 |
|  | 8 | 13.5 | 22.2 | 23.1 | 24.9 | 23.1 | 25.5 | 25.4 | 26.3 |
|  | 11 | 11.9 | 20.3 | 22.7 | 25. 0 | 23.6 | 25.8 | 25.2 | 26.7 |
|  | 18 | 20.9 | 24.6 | 24.9 | 25.3 | 24.3 | 25.9 | 26.5 | 26.4 |
|  | 35 | 13.0 | 19.9 | 21.3 | 23.6 | 25.1 | 24.8 | 25.3 | 25.6 |
| TMI-2. | 5 | 13.3 | 19.3 | 20.9 | 22.4 | 22.4 | 23.7 | 23.3 | 24.0 |
|  | 19 | 17.6 | 22.4 | 22.8 | 23.4 | 23.1 | 24.0 | 23.7 | 24.3 |
|  | 26 | 11.1 | 17.9 | 20.0 | 22.7 | 23.0 | 25.0 | 24.9 | 25.4 |
| TR-1 | 9 | 16.8 | 21.3 | 21.5 | 22.1 | 21.5 | 22.8 | 22. 4 | 23.3 |
| TR-2 | 6 | 14.7 | 20.1 | 21.2 | 22.4 | 22.0 | 22.8 | 22.3 | 23.5 |
|  | 10 | 13.6 | 19.1 | 19.5 | 19.8 | 19.8 | 20.6 | 20.0 | 21.3 |
| TH-1 | 27 | 15.3 | 19.4 | 20.9 | 22.1 | 21.2 | 22.6 | 22.6 | 22.7 |
|  | 2 | 7.6 | 12. 5 | 13.7 | 15. 2 | 14.2 | 15.8 | 15.4 | 16.2 |
|  | 12 | 4.1 | 8.4 | 11.0 | 13.4 | 12.3 | 14.1 | 14.6 | 14.7 |
|  | 32 | 2. 0 | 8.2 | 10.8 | 14.3 | 15. 0 | 18.2 | 19.9 | 19.7 |
|  | 28 | 4. 2 | 8.8 | 10.7 | 12.8 | 12.6 | 14.0 | 15.0 | 14.5 |
| TP-1 | 13 | 2.3 | 5.3 | 7.3 | 8.3 | 7.1 | 8.9 | 9.4 | 9.9 |
| TP-2 | 29 | 2. 7 | 6.4 | 7.9 | 9.8 | 10.4 | 11.4 | 10.6 | 11.7 |
| TP-4 | 4 | 2. 7 | 5.3 | 6. 4 | 7.7 | 7.4 | 8. 2 | 7.6 | 8.7 |
| TP-6 | 30 | 1. 2 | 4.3 | 5.3 | 6.5 | 6.8 | 7.5 | 7.1 | 8.2 |
| Special | 34 | - 7 | 3.4 | 4. 8 | 7.2 | 8.7 | 9.7 | 9.6 | 10.5 |
|  |  | 7.3 | 12.2 | 13.5 | 14.9 | 14.2 | 15. 7 | 15. 3 | 16.0 |
|  | $23$ | 29.9 | 41.4 | 44.4 | 45.4 | 46. 6 | 46. 1 | $4 \times .4$ | 46.3 |
|  | 31 | 2.9 | 6.2 | 6.4 | 7.0 | 6.7 | 7.8 | 6.0 | 8.1 |

During exposure a skin was formed on the surface of the samples, the thickness of the skin increasing as the loss of the volatile constituents increased. The thickness of the skin had increased noticeably by the end of each exposure period, the depth of softer material on the bottom of the pan being correspondingly reduced. Photographs showing the condition of the film surfaces after 15 weeks exposure are shown in figures 8 to 11 inclusive.

It will be noticed from table 12 and figure 7 that all of the tars lost weight rapidly at first and then more slowly as the time of exposure increased. Based on the loss after 15 weeks, the maximum loss in 2 days was 79.2 percent, the minimum 6.7 , and the average loss for the 32 tars meeting some specification was 51.7 percent. In 15 days the maximum loss was 95.6 , the minimum 45.7 , and the average 84.1 percent. Beyond the 15 -day period all samples lost weight very slowly. At the end of the 50 -day period the average loss for all samples was 97.4 percent of the loss at 15 weeks. The cold-application tars showed a much higher rate of loss than did the hot-application tars. At the end of 8 days the former showed an average loss of 86.1 percent of


Figure 6.- Exposure Boxes Containing Tar samples and Hubbard-Field Spectmens During an Kxposure Test.


Figure 7.-Rate of Loss in Thin-Film Exionure of Variouis
Road Tars (Values Are Averages for Each Crade)
their ultimate loss, while the nine hot tars required a period of 22 days to show an average loss of 83.2 percent.

In order to make comparisons of the characteristics developed by 5 -, 10 -, and 15 -week exposures and by the distillation test, the respective residues were subjected to the same tests. These data are shown in table 13.

EXPOSURE RESIDUES WERE HARDER AND DEVELOPED MORE FREE CARBON THAN DISTILLATION RESIDUES
The average total loss in the distillation test, A. S. T. M. D 20-30, for the 32 tars that substantially met the Federal specifications was 25.3 percent as compared to the average loss of the same samples for 5,10 , and 15 -week exposure periods of $22.3,23.2$, and 24.0 percent, respectively. Since the average total loss in the distillation test, when the residue was poured immediately, was 27.5 percent for these same samples, it is seen that the ultimate loss as determined at the end of the 15 -week period was in closer agreement with the loss obtained in the standard method of distilling tar products. Figure 12 compares the total loss in the distillation test, A. S. T. M. D $20-30$, with the loss occurring in thin films at the end of 15 weeks of exposure. Samples 32 and 34 were the only tars to show an appreciably greater loss under exposure than occurred in the distillation test. All other materials had losses approximately equal to or less than the losses in distillation.

Although the loss of volatile matter in 5 weeks was practically equal to or less than the loss in the distillation test, except for samples 32 and 34 , all the residues from this period except for samples, $9,10,22$, and 31 , had penetrations equal to or lower and softening points


Figure 8.-Appearance of Surfaces of Tar Samples After 15 Weers of Exposure. The Figures in Parentheses Are the Federal Specification Grades to Which the Samples Conform. The Centers of the Spots on Samples 7 and 1 Were Highly Carbonized. All of the Other Sampifes whre Slightly Carbonized.
equal to or higher than the corresponding distillation residues.

The greatest diflerence in penetration occurred in the exposure residues of samples $23,24,26,32,33,34$, and 35 , their penctrations ranging from one-ninth to one-twenty-fifth of the penctrations of their corresponding distillation residues. At the end of 10 weeks only the residue of sample 31 had a softening point appreciably
lower than that of its distillation residue. The softening point of the exposure residue of sample 22 was approximately the same as that of its distillation residue. After 15 weeks of exposure all residues, except that of sample 31, were considerably harder than their corresponding distillation residues.

A comparison of the softening points and penetrations of the distillation residues and the 15 -week exposure



SAMPLE 8. (TM-1)


SAMPLE 11. (TM-1)


SAMPLE 18.(TM-1)

SAMPLE 35.(TM-I)


SAMPLE 5. (TM-2)


SAMPLE 19. (TM-2)

Figure 9.--Appearance of Surfaces of Tar Samples After 15 Weeks uf Exposure. The Figures in Parenthesbes Are the Federal Specification Grades to Whicif the Samples Conform. The Center of the Spot on Sample S Wia Hifhly Carbonized and the Rough Arbas on Sample 11 Werb Carbonizfd. Abi of the Other Samplas Werki

residues substantiates the conclusion of Reeve and Anderton that the comparative consistencies of the residues from distillation to a single arbitrary temperature do not represent the relative behavior of tars in service. The average softening point of the distillation residues of samples $7,14,20$, and 24 meeting $\mathrm{TC}-1$ specification was $48.1^{\circ} \mathrm{C}$., and the average softening point of the
residues after 15 weeks of exposure was $74.5^{\circ}$ (? The distillation residues of the five TP products, samples 4, $13,29,30$, and 34 , had an average softening point af $50.1^{\circ} \mathrm{C}$., but the average softening point of their exposure residues was slightly under $65^{\circ}$ ()

Early investigators, as previously mentioned, noted hat tar products on exposure developed considerable


Figure 10.- Appearance of Surfaces of Tar Samples After 15 Weeks of Exposure. The Figures in Parentheses Are The Federal. Specification (irades to Wheh the Samples Conform. The Concentric Circles on Sample 12 Show Increasing (Carbonization Toward the Center, and the Spots on Samples 9 and 10 Where Carbonized. All of the

quantities of free carbon. In order to see if this produrtion of organie material was more pronomenced under exposure conditions than under the aceelerated laboratory heat test (the distillation test), determinations of solubility in curbon disulphide were made on all residues thus obtamed. By expressing the percentage of free curbon in any residue in terms of the weight of the
original material, then dividing by the percentage of free carbon in the original material and multiplying by 100 , the result is the index of increase in free carbon. An index of 100 indicates that no increase nor decrease has taken place. 'The indexes of increase in free carbon for the various residues are given in table 14 .



 Carbonized. In Amdition, samples 29 and 30) Were Shfihtly Fldorescent.

LOSS OF VOLATILE MATTER, OXIDATION, ANID POLYMERIZATION CAUSE CHANGES UPON EXPOSURE
This table indicates that the distillation residues of samples $6,7,8,9,10,11,12,1: 3$, and $2: 3$ and the residues from exposure of sample 7 at 5 and 10 weeks and of sample 9 at 15 weeks had indexes of increase of less than
100. These apparent decreases in free carbon may, in the majority of cases, result from slight maecuracies in the carbon-disulphide solubility tests. In the case of sample 23, repeated check tesits on both the original material and the distillation residue indieated that the free carbon content of this material was greatly reduced

figure 12.- ('omparison of the 15-Week Exposure Loss With the Total Loss in Distillation.
duriner distillation. This sample under exposure rapidly developed material insoluble in carbon disulphide. For all materials, except samples 7 and 31, the index of increase in free carbon is least in the distillation residues and greatest in the 10 - or 15 -week exposure residues.

As measured by the softening point, the hardness of the 5 -week exposure residues of samples $1,20,23,24$, $25,26,32,33,34$, and 35 exceeded the hardness of their distillation residues by the greatest amounts, the difference in softening points ranging from $14.4^{\circ} \mathrm{C}$. to

Table 14.-Index of increase in free carbon for various residues

| Type of material | Identification | Distillation residue | Exposure residues |  |  |
| :---: | :---: | :---: | :---: | :---: | :---: |
|  |  |  | 5 weeks | 10 weeks | 15 weeks |
| TC-1$\mathrm{TC}-2$ | 7 | Percent | Percent | Pcrcent | Pcrcent |
|  | 14 | 116 | 131 | 15.4 | 184 |
|  | 20 | 172 | 262 | 294 | 328 |
|  | 24 | 126 | 194 | 210 | 220 |
|  | 1 | 100 | 152 | 169 | 169 |
|  | 15 | 120 | 139 | 162 | 155 |
|  | 21 | 170 | 258 | 287 | 302 |
|  | 33 | 121 | 216 | 245 | 339 |
| TC-3_ | 16 | 122 | 140 | 163 | 172 |
|  | 125 | 122 | 185 | 190 | 205 |
| TC-4 | $\{3$ | 103 | 133 | 156 | 163 |
| TM-1 | 17 | 130 | 148 | 162 | 172 |
|  | -888 | 96 | 118 | 126 | 129 |
|  | 11 | 93 | 120 | 130 | 133 |
|  | 18 | 125 | 148 | 166 | 178 |
|  | 35 | 123 | 189 | 202 | 240 |
| TM-2 | 5 | 104 | 138 | 142 | 152 |
|  | 19 | 126 | 145 | 164 | 168 |
|  | 26 | 119 | 176 | 193 | 201 |
| TR-1 | 9 | 88 | 109 | 110 | 94 |
| TR-2 | 6 | 93 | 109 | 113 | 117 |
|  | 10 | 93 | 112 | 111 | 120 |
| TII-1 | 27 | 116 | 132 | 143 | 145 |
|  | 2 | 101 | 122 | 122 | 128 |
|  | 12 | 97 | 120 | 134 | 131 |
| TIT-1 | 32 | 112 | 191 | 205 | 237 |
|  | 28 | 121 | 153 | 169 | 173 |
| TP-1 | 13 | 98 | 119 |  | 129 |
| TP-2 | 29 | 120 | 144 | 149 | 161 |
| TP-1 | 4 | 112 | 121 | 118 | 127 |
| ' ${ }^{\text {P-6 }}$ | \{ 30 | 113 | 132 | 136 | 1.50 |
| Special | 34 | 110 | 162 | 176 | 18: |
|  | 22 | 115 | 118 | 122 | 127 |
|  | 23 | 14 | 210 | 188 | 214 |
|  | 31 | 128 | 123 | 131 | 111 |

$26.4^{\circ} \mathrm{C}$. The softening points of the 15 -week exposure residues of these same materials exceeded the softening points of the distillation residues by amounts ranging from $21.9^{\circ} \mathrm{C}$. to $41.4^{\circ} \mathrm{C}$. With the exception of sample 21, the difference between the index of increase in free carbon for the exposure and distillation residues was also greatest for these materials. For the 5 -week

TABLE 13.-Results of laboratory tests on distillation and exposure residues

| Type of material | Identification | Distillation residue (A. S. T. M. D 20-30) |  |  |  | 5-week exposure residue |  |  |  | 10-week exposure residue |  |  |  | 15-week exposure residue |  |  |  |
| :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: |
|  |  | Total loss | $\begin{gathered} \text { Pene- } \\ \text { tration } \\ \text { at } 25^{\circ} \\ \text { C., } 100, \\ \text { g., } 5 \text { sec. } \end{gathered}$ | Softening point | Free carbon | Loss | Penetration at $25^{\circ}$ C., 100 , g., 5 sec . | Softening point | Free carbon | Loss | Penetration at $25^{\circ}$ C., 100, g., 5 sec | Softening joint | Free carbon | Ioss | Penetration at $25^{\circ}$ C., 100 , g., 5 sec. | Softening point | Free carbon |
| T ${ }^{\prime}-1$ |  |  |  |  | Percent | Percent |  |  | Percent | Percent |  |  | Percent | Percent |  | ${ }^{\circ} \mathrm{C}$. | Percent |
|  | 7 | $35.90$ | 38 | 45.0 | 11.7 | 33.2 | 15 | 58.0 | $9.8$ | 34.4 | 5 | 69.4 | 11.6 | 34.0 | 2 | 73. 3 | 14.4 |
|  | 14 | 38.04 | 25 | 54.8 | 11.4 | 33.1 | 13 | 61.0 | 11.9 | 33.7 |  | 69.2 | 14.1 | 33.6 | 2 | 74.3 | 16.9 |
|  | 20 | 32.06 | 38 | 47.4 | 11.9 | 33.9 | 6 | 62.9 | 18.6 | 33.3 | 2 | 68.7 | 20.7 | 34.5 | 1 | 74.2 | 23.5 |
|  | 24 | 34.35 | 50 | 45.2 | 12.6 | 35.3 | 2 | 66. 7 | 19.8 | 35.3 |  | 70.8 | 21.4 | 36.3 | 1 | 76.3 | 22.8 |
|  |  | 33. 19 | 38 | 41.6 | 9.1 | 31.4 | 9 | 59.3 | 13.5 | 31.1 | 5 | 66.7 | 15.0 | 32.3 | 2 | 71.9 | 15. 2 |
|  | 15 | 36. 34 | 20 | 55. 6 | 12.2 | 30.5 | 19 | 57.9 | 12.9 | 30.7 | 7 | 65.0 | 15.1 | 31.7 | 4 | 70.4 | 14.8 |
|  | 21 | 29. 25 | 27 | 50.8 | 13.0 | 29.2 | 6 | 61.1 | 19.6 | 29.1 |  | 66.8 | 21.8 | 30.7 | 1 | 75.2 | 23.5 |
| T ${ }^{*}-3$ | 33 | 29.92 | $13 \times$ | 37.0 | 6. 6 | 30.8 | 7 | 59.4 | 11.8 | 32.7 |  | 67.3 | 13.8 | 30.5 | 2 | 71.5 | 18.6 |
|  | 16 | 35. 50 | 18 | 57.1 | 12.8 | 28.8 | 16 | 59.5 | 13. 4 | 29.5 | 6 | 68.0 | 15.7 | 30.9 | 3 | 75.9 | 17.0 |
|  | 2.5 | 31.89 | 42 | 46.6 | 12.9 | 31.6 | 3 | 64.9 | 19.4 | 31.3 |  | 67.3 | 19.9 | 32.7 | 1 | 74.2 | 22.0 |
| 'I' $\quad \cdots 4$ | $\left\{\begin{array}{r}3 \\ 17\end{array}\right.$ | 29. 10 | 53 | 45.0 | 11.2 | 26.2 | 12 | 56.9 | 13.5 | 23.4 | 2 | 68.8 | 15.3 | 28.6 | 2 | 71.3 | 17.1 |
|  | $\begin{array}{r}17 \\ 8 \\ \hline\end{array}$ | $\begin{aligned} & 34.78 \\ & 26.05 \end{aligned}$ | 12 | 60.4 41.0 | 13.8 | 26.0 | 17 | 59.0 51.3 | 13.6 13.4 | 27.1 25.4 |  | 68.5 62.8 | 15.3 | 28.4 | 5 | 74.2 66.4 | 16.6 |
| TM-1. | 11 | 27. 57 | 74 | 42.0 | 11.0 | 23.6 | 35 | 50.2 | 13.5 | 25.2 | 15 | 59.3 | 14.9 | 26. 7 | 6 | 65.4 | 15.2 15.6 |
|  | 18 | 33. 42 | 15 | 59.4 | 13.6 | 24.3 | 15 | 60.0 | 14.3 | 26.5 | 5 | 70.6 | 16.5 | 26.4 | 2 | 74.9 | 17.6 |
|  | 35 | 25. 31 | 97 | 39.6 | 7.8 | 25.1 | 9 | 57.5 | 11.9 | 25. 3 |  | 61.6 | 12.7 | 25.6 | 4 | 64.5 | 15.2 |
| TM-2.. | 5 | 25. 73 | 46 | 46.1 | 11.4 | 22.4 | 16 | 54.5 | 14. 6 | 23.3 |  | 61.1 | 15.1 | 24.0 | 3 | 65.4 | 16.4 |
|  | 19 | 3189 | 12 | 60.0 | 13.7 | 23.1 | 14 | 59.7 | 13.9 | 23.7 | 8 | 65.7 | 15.8 | 24.3 | 4 | 68.4 | 16.4 |
|  | 26 | 23. 40 | 62 | 43.2 | 13.0 | 23.0 | 7 | 60.0 | 19.2 | 24.9 |  | 67.7 | 21.6 | 25.4 | 2 | 71.8 | 22.6 |
| TR-1 | 9 | 27. 75 | 12 | 60.0 | 14.3 | 21.5 | 25 | 55.5 | 16.3 | 22.4 |  | 62.9 | 16.6 | 23.3 | 6 | 68.0 | 14. 4 |
| T R-2 | 6 | 25. 90 | 20 | 53.1 | 20.3 | 22.0 | 12 | 57.8 | 22.5 | 22.3 | 7 | 63.1 | 23. 6 | 23.5 | 3 | 67.5 | 24.7 |
|  | 10 | 25.52 | 18 | 55.8 | 11.4 | 19.8 | 27 | 54.0 | 12.8 | 20.0 | 10 | 59.5 | 12.8 | 21.3 | 6 | 67.0 | 14.0 |
|  | 27 | $25.45$ | 15 | 55.4 | 17.5 | 21. 2 | 10 | 58.3 | 18.9 | 22.6 |  | 65.1 | 20.9 | 22.7 | $\stackrel{2}{2}$ | 66.4 | 21. 2 |
| T $11-1$ | 2 | 19.80 | 26 | 51.7 | 18.7 | 14. 2 | 15 | 56.0 | 21. 1 | 15. 4 | 4 | 64. 1 | 21.4 | 16.2 | 2 | 67.4 | 22.7 |
|  | 12 | 15. 54 | 89 | 41.6 | 11.7 | 12.3 | 49 | 47.6 | 13.9 | 14.6 | 18 | 57.6 | 15.9 | 14.7 | 15 | 59.0 | 15.7 |
|  | 32 | 11. 12 | 218 | 33.1 | 8. 6 | 15.0 | 17 | 53.0 | 15.3 | 19.9 |  | 65.7 | 17.4 | 19.7 | 2 | 68.7 | 20.1 |
|  | 28 13 | 14.37 | 43 38 | 47.0 | 13.7 | 12.6 | 13 | 57.0 | 16.9 | 15. 0 |  | 64.7 | 19.3 | 14.5 | 2 | 65.2 | 19.7 |
| $\begin{aligned} & \mathrm{TP}-1 . \\ & \mathrm{TP}-2 \end{aligned}$ | 13 | 11. 79 | 38 | 49.0 | 11.9 | 7.1 | 41 | 49.3 | 13.7 | 9.4 | 11 | 61.4 |  | 9.9 | 8 | 63.4 | 15.2 |
| TP-2 | 29 | 11. 72 | 30 | 49.6 | 15.2 | 10.4 | 5 | 59.9 | 18.0 | 10.6 |  | 63.1 | 18.7 | 11.7 | 3 | 66.6 | 20.4 |
| TP-4 | 4 | 13. 20 | 12 | 56.1 | 21.1 | 7.4 | 13 | 56.4 | 21.4 | 7.6 | 8 | 60.7 | 21.0 | 8.7 | 2 | 64.0 | 22.9 |
| 'TP-6 | 30 | 8. 72 | 25 | 51.8 | 15.1 | 6. 8 | 12 | 56.5 | 17.3 | 7.1 |  | 59.5 | 17.9 | 8.2 | 4 | 64.0 | 19.9 |
| Special | 34 | 5. 90 | 71 | 44.1 | 9.2 | 8.7 | 5 | 58.5 | 14.0 | 9.6 |  | 62.9 | 15.4 | 10.5 | 2 | 66.0 | 16.6 |
|  | $22$ | $19.62$ | 15 | 58.5 | 30. 1 | 14. 2 | 40 | 50.0 | 29.0 | 15.3 |  | 57.6 | 30. 3 | 16.0 | 5 | 63.0 | 31.9 |
|  | 23 | $47.85$ | 84 | 39. 1 | 1.3 | 46.6 | 6 | 65.5 | 19.6 | 48.4 |  | 77.6 | 18.2 | 46.3 | 1 | 80.5 | 20.0 |
|  | 31 | 18.90 | 1 | 73.3 | 20. 1 | 6.7 | 42 | 51.7 | 16. 7 | 6.0 |  | 59.4 | 17.7 | 8.1 | 3 | 68.5 | 19.5 |

exposure residues the difference ranged from 52 to 196 and for the 15 -week exposure residucs the difference ranged from 69 to 218.
It was shown by Reeve and Anderton ${ }^{10}$ that watergas tars low in free carbon increased in free-carbon content to a much greater extent than coal tars high in free carbon when exposed under conditions comparable to those of this investigation. Because of the large number.of blended tars and the wide range in consistency of the materials covered in this study, the relations between free carbon in the original materials and the freecarbon content of the residues after exposure only indicate a general trend that the lower the free-carbon content of the original material the higher the index of free carbon in the exposed residue. This trend is shown by table 15, which is based on the test data given in tables 5 and 14.
S. Sabrou, ${ }^{13}$ in reporting on a study of the aging of road tars, drew the following conclusions, based on laboratory studies. When a tar is spread on a road and subjected to the climatic conditions prevailing in France, it undergoes an aging which is not attributed to polymerization due either to heat or the ultra violet light of the sun, but which is attributed to slow and slight oxidation and above all to the evaporation of the lighter constituents. He intimated that the effect of evaporation is at least 100 times that of oxidation.
Some of the inherent changes developed on exposure can probably be traced to the high loss of volatile matter which occurred early in the exposure period, thereby causing a considerable reduction in film thickness. This reduction in film thickness undoubtedly caused the material to weather more rapidly. An example of this is shown by the test results on the four samples submitted by producer E. These data are shown in table 16.
Since these materials are probably from the same base, they may be assumed to be comparable. The figures show that as the loss of volatile material increases with attendant decrease in film thickness, the index of increase in free carbon also increases. Thus it is indicated

Table 15.-Comparison of free carbon content of original material and index of increase in free carbon for exposure residues

| Number of samples | Free carbon in original materials |  |  | Index of increase in free carbon for residues after 15 weeks' exposure |  |  |
| :---: | :---: | :---: | :---: | :---: | :---: | :---: |
|  | Maximum | Minimum | A verage | Maximum | Minimum. | A verage |
|  | Percent | Percent | Percent |  |  |  |
| 12 |  |  |  | ${ }_{237}^{339}$ | 155 | 285 |
| 12 | 11.67 | 8.15 | 9. 68 | 201 | 94 | 141 |
| 6. | 21. 09 | 12. 23 | 15. 57 | 150 | 117 | 132 |

Table 16.-Results of tests on the 15 -week exposure residues of samples obtained from producer $E$


[^9]Table 17.-Comparison of averaged ${ }^{1}$ test resulls on distillation and exposure residues

| 'Test | Average total loss | Average penetration of residue | Average softening point of residue | Average inder of inerease itl frec earhon |
| :---: | :---: | :---: | :---: | :---: |
| Distillation | Percent 25. 3 |  | ${ }^{-} C_{49.0}$ | 111 |
| 5-week exposure | 22.3 | 15.5 | 57.5 | 1.50 |
| 10-week exposure | 23.2 |  | 64.9 | 164 |
| 15-week exposure. | 24.0 | 3.3 | 69.1 | 176 |

1 Data for samples 22, 23, and 31 excluded.
that the more viscous tars, which lose less volatile material and produce greater film thicknesses, should weather less rapidly than the lighter tars.

A general comparison of the effects of exposure as compared to the effects produced by the distillation test may be clearly seen in table 17. This table shows that the 5 -week exposure residues are considerably harder than their corresponding distillation residues.

The continued increase in hardness and the increase in free-carbon content of the residues exposed for longer periods without much additional loss of volatile matter indicate that the changes in the character of the residues after exposure are due to other causes than mere loss of volatile matter. There is evidence that both oxidation and carbonization are responsible for the changes that take place when bituminous materials are exposed to air and sunlight. The great increase in the freecarbon content of the tars subjected to exposure, as well as the greater hardness of the exposed residues, indicates quite clearly that mere evaporation is not responsible for the highly altered residues obtained.

HUBBARD-FIELD STABILITY TEST USED TO DETERMINE BONDING: STRENGTH BEFORE AND AFTER EXPOSURE
This and previous investigations have shown that on exposure tars develop free carbon in excess of that caused solely by concentration. No efforts have been made to determine the character of this free carbon. G. T. Gilbert and J. G. Mitchell ${ }^{14}$ have stated that quantitative determinations show that tars oxidize in the dark and that this oxidation is accelerated by light with the formation of material insoluble in benzenc (free carbon). These writers have advanced a new conception of tars based on the solubility of tar in various solvents. They indicate that free carbon is of four types: $\mathrm{C}_{1}$, insoluble in all solvents; $\mathrm{C}_{2}$, insoluble in benzol $\left(\mathrm{C}_{6} \mathrm{H}_{6}\right)$ but soluble in pyridine; $\mathrm{C}_{3}$, insoluble in ethyl ether but soluble in benzene; and $\mathrm{C}_{4}$, insoluble in light naphtha ( $40^{\circ}-60^{\circ} \mathrm{C}$. boiling point) but soluble in ether. They intimated that the formation of this benzene-insoluble free carbon can be detected by determining the $\mathrm{C}_{1}$ and $\mathrm{C}_{2}$ content of the original and exposed tars. The ratio $\frac{\mathrm{C}_{1}+\mathrm{C}_{2}}{\mathrm{C}_{1}}$ then gives a measure of increase distinct from that caused by simple concentration resulting from evaporation. The character of the free carbon formed in exposed tars, based on the solubility of the original material and the residue from exposure in benzene or carbon disulphide and pyridine, may prove of value in detecting the internal alterations that accompany the weathering of tars.

Road tars when used in the road surface develop adhesiveness or binding strength in various ways de-

[^10]pending upon the grade of tar and the type of construction in which it is used. The tars that are used in penetrative treatments such as primers, those used as binders for cover stone in surface treatments and seal coats, and those used as binders for coarse open-graded mixtures, either plant or road mixed, and in some cases for closely graded mixtures, all develop their initial binding strength through the loss of their more volatile constituents, or through stiffening of the material on cooling, or a combination of both.

Cold-application tars have little bonding strength when first applied to the road, but by rapid loss of volatile matter soon provide a very stable surface. Hot-application tars of both the surface treatment and construction grades possess sufficient bonding strength when cooled in the road surface to withstand immediately the stress of traffic, although the initial bonding strength of the hot-surface-treatment tars may be further increased by the loss of volatile matter. The ultimate bonding strength of all road tars is undoubtedly increased by a continued loss of volatile matter and by inherent changes when exposed to service conditions.

In order to determine the initial bonding strength and its development, a series of tests were made using the Hubbard-Field stability apparatus. This equipment has been extensively used to study the effects of various physical properties of asphaltic materials on the stability or resistance to displacement of fine-graded asphaltic mixtures. ${ }^{15}$

Determinations of the bonding strength or adhesiveness of the 35 tars were made by preparing HubbardField stability cylinders containing 16.6 percent by volume of original materials and their residues from distillation and 83.4 percent of a standard sand. These molded specimens are designated as series 1 cylinders.
The development of bonding strength was determined by preparing cylinders using the same percentage of original materials and sand as stated above and exposing the compressed specimens to the same conditions as the thin films. For comparative purposes cylinders were prepared using the amount of each distillation residue that would have been present had the percentage of tar in the cylinders containing the original material been subjected to the distillation test. These specimens, together with the exposed cylinders are designated as series 2 cylinders.

The aggregate used in making these tar mixes was the same Potomac River sand used in the previous work on liguid asphaltic mixes. ${ }^{12}$ The sand was washed, separated into various sizes, and recombined to give the following grading:

|  | Percent |
| :---: | :---: |
| Passing sieve no. 20, retained on sieve no. 30 | 0. 3 |
| Passing sieve no. 30, retained on sieve no. 40 | 18.1 |
| Passing sieve no. 40 , retained on sieve no. 50 | 21. 3 |
| Passing sieve no. 50, retained on sieve no. 80 | 36.6 |
| Passing sieve no. 80, retained on sieve no. 100 | 6.1 |
| Passing sieve no. 100 , retained on sieve no. 200 |  |
| Passing sieve no. 200 |  |

The tar products and sand were thoroughly mixed by hand. Three cylinders were prepared for each test condition. All cylinders were compacted under a load of 3,000 pounds per square inch. Since the 35 tars

[^11]varied greatly in consistency, the test procedure for making and testing the Hubbard-Field specimens varied as follows:

1. The original materials intended for cold application were mixed and compressed at room temperature.
2. The original materials intended for hot application were mixed and compressed at $40^{\circ} \mathrm{C}$.
3. All distillation residues were mixed and compressed at $80^{\circ} \mathrm{C}$.
4. In forming cylinders containing the original materials, the pressure was released as soon as the required total load was reached. For the cylinders containing the distillation residues the total load was held for 2 minutes, and the mold was flooded with cold water to chill the specimens.
5. The bonding strength or stability of the cylinders containing the original materials was determined at $25^{\circ} \mathrm{C}$. only. The cylinders from the 5 -week period of exposure were tested for stability at $25^{\circ} \mathrm{C}$. and, if the total load was over 10,000 pounds on the first specimen, the other two specimens were tested at $60^{\circ} \mathrm{C}$. The cylinders containing the distillation residues were tested at $60^{\circ} \mathrm{C}$. except in a few cases where one of the three cylinders was tested at $25^{\circ} \mathrm{C}$. The stabilities of the cylinders exposed for 10 and 15 weeks were all determined at $60^{\circ} \mathrm{C}$.

EXPOSED CYLINDERS HAD MUCH HIGHER STABILITIES THAN CYLINDERS MADE WITH DISTILLATION RESIDUES
The results of the stability tests on cylinders of series 1 are given in table 18, and those on series 2 are given in table 19. These data have been plotted in figures 13,14 , and 15.
In figure 13 the stability of the cylinders made with the cold-application tars has been plotted against

Table 18.-Results of tests on series 1 cylinders

| Type of material | Identification | Original material |  |  | Distillation residue ${ }^{1}$ |  |  |  |
| :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: |
|  |  | $\begin{aligned} & \text { Stabil- } \\ & \text { ity at } \end{aligned}$$25^{\circ} \mathrm{C}$ | Specific <br> viscosity at $40^{\circ} \mathrm{C}$. | $\begin{aligned} & \text { Float } \\ & \text { at } \\ & 50^{\circ} \mathrm{C} . \end{aligned}$ | Stability |  | Softening point | Penetration at $25^{\circ} \mathrm{C}$. |
|  |  |  |  |  | $\begin{aligned} & \text { Tested } \\ & \text { at } \\ & 60^{\circ} \mathrm{C} . \end{aligned}$ | Tested at $25^{\circ} \mathrm{C}$. |  |  |
| TC-1. |  | Pounds |  | Seconds | Pounds | Pounds | $\bigcirc$ C. |  |
|  |  | 0 | 9. 2 |  | 800 |  | 54.0 | 21 |
|  | 14 | 0 | 10.7 9.5 |  | 1,475 850 |  | 61.7 | $\stackrel{9}{12}$ |
|  | 24 | 0 | 11.8 |  | 925 |  | 54.4 | 13 |
| TC.2 | 1 | 15 | 15.9 |  | 513 | 8, 100 | 50.1 | 30 |
|  | 15 | 10 | 15.5 |  | 1,513 |  | 62.7 | 8 |
|  | 21 | 2.5 | 17.3 |  | 790 |  | 57.0 | S |
|  | 33 | $(0)$ | 16.2 |  | 398 |  | 58.1 | 36 |
| TC-3 | 16 | 63.3 | 21.2 |  | 1,617 |  | 63.1 | 7 |
|  | 25 | 70 | 20. 7 |  | 585 | $10,000-+$ | 52.2 | 19 |
| TC-4 | 3 | 87 | 34.9 | -.. | 650 |  | 54. 0 | $1 i^{\prime}$ |
|  | 17 | 90 | 32.5 |  | 1, 833 |  | 64. 4 | 5 |
| TM-1 | 8 | 100 | 40.1 |  | 525 | 6, 050 | 48.4 | 34 |
|  | 11 | 80 | 53.5 |  | 538 | 6,900 | 49.2 | 41 |
|  | 18 | 90 | 48.5 |  | 1,567 |  | 64.1 | 6 |
|  | 35 | 117 | 57.4 |  | 375 | 9,100 | 48.2 | 36 |
| TM-2 | 5 | 153 | 70.4 |  | 608 | .......- | 52.5 | 18 |
|  | 19 | 133 | 70.6 |  | 2, 030 |  | 65.8 | 5 |
|  | 26 | 173 | 83.7 |  | 488 | 9,800 | 50.7 | 25 |
| TR-1 | 9 | 153 | 44.5 |  | 1,892 |  | 65.0 | 7 |
| TR-2 | 6 | 178 | 60.7 |  | 1,150 |  | 60.4 | 5 |
|  | 10 | 153 | 73. 1 |  | 1,260 |  | 61.0 | $?$ |
|  | 27 | 163 | 63.1 |  | 1,010 |  | 59.5 | 1 |
| TH-1 | 2 | 580 |  | 58 | 1, 110 |  | 58.1 | 9 |
|  | 12 | 380 |  | 51 | - 475 | 5, 800 | 44.8 | 67 |
|  | 32 | 310 |  | 43 | 225 | 4,200 | 39.7 | 98 |
|  | 28 | 783 |  | 78 | 550 |  | 52.8 | 21 |
| TP-1 | 13 | 1,200 |  | 107 | 800 |  | 54.0 | 22 |
| TP-2 | 29 | 1,958 |  | 126 | 883 |  | 56.0 | 10 |
| TP-4 | 4 | 2,842 |  | 167 | 1,375 |  | 60.1 | 5 |
| TP-6. | 30 | 3,417 |  | 194 | 800 |  | 56.8 | 12 |
| Special | 34 | 2,258 |  | 165 | 625 |  | 54.4 | 13 |
|  | 22 | 310 |  | 30 | 1,700 |  | 61.6 | 6 |
|  | 23 | 0 | 2.0 |  | 892 |  | 52.7 | 26 |
|  | 31 | 1,360 |  | 51 | 4,867 |  | 75.6 | 1 |

[^12]

Figure 13.-Relations Between the Consistencies of Original Materials and the Stability at $25^{\circ} \mathrm{C}$. of Series 1 Cylinders.


Figure 14.-Relations Between Consistenciles of the Distillation Residues and Stability at $60^{\circ} \mathrm{C}$. of Series 1 Cylinders.
the Engler specific viscosity at $40^{\circ} \mathrm{C}$. of the contained tar, and for the cylinders made with the hot-application tars the stability is plotted against the float test in seconds at $50^{\circ} \mathrm{C}$. The stability values ob tained on the cylinders containing the cold-application materials


Figure 15.-Comparison of Loss of Bitumen and stablith of Serifes 2 Hubbard-Field Cylinders.
are low since the viscosity range indicates relatively fluid products. The greater consistency of the hoiapplication materials results in higher stability values. Generally, with both types of material the higher the consistency of the tar used as binder the higher was the stability. A comparison of the two curves clearly illustrates the difference in binding ability of cold- and hot-application materials immediately after being applied. The cold-application materials must rapidly lose much of their volatile constituents to increase the stability of the road surface.

The stability of the cylinders containing the distillation residues was plotted against the softening point and penetration of the contained tar pitch, as shown in figure 14. The softening point value showed a more constant relation than did the penetration value, indicating that the softening point value of a tar pitch is the better measure of its relative bonding strength. The relation between stability and penetration did

Table 19.-Results of tests on series 2 cylinders

show, however, that for equal penctrations the watergas tar residues generally gave higher stabilities than the residues from coal tars or coal-tar blends.

The Hubbard-Field stability values for the series 2 cylinders are shown in figure 15 , along with the percentages of volatile matter lost under test conditions. Although in the cylinders made with the original materials the loss of volatile matter after exposure was very close to the loss in the distillation test, the stabilities of the cylinders exposed for 15 weeks were much greater than the stabilities of the cylinders prepared with the distillation residues. This shows that changes other than loss of volatile matter have produced a condition in the mixture that results in a greater bonding strength, just as greater hardening and internal alterations in the exposed films tended to produce residues dissimilar to those developed in the laboratory distillation tests.

The loss of volatile matter for the cylinders exposed for 10 weeks is not shown in figure 15 since it closely approximated the loss at 15 weeks. But figure 15 shows that in the cylinders of many materials there were increases in stability from 10 to 15 weeks that may be attributed to additional hardening or change in the contained tar residue without corresponding loss of volatile matter.
W. E. Cone has stated ${ }^{16}$ that: "The ease and facility with which tar wets a particular surface and retains the full value of its adhesive properties while being gradually changed to a solid is one of its most striking characteristics."

[^13]While testing exposed cylinders that initially contained liquid asphaltic material even of the rapidcuring type, it was observed that while a hardened crust formed, which in some cases was very thick, the mixture in the center of the test specimens was plastic after 15 weeks of exposure. The exposed cylinders containing these tar products, in many cases after 5 weeks of exposure and in all cases after 15 weeks, appeared to be uniformly hard and solid throughout the entire mass, indicating that ultimate hardening of the contained tar is not materially retarded by the formation of a surface crust.

Some idea of the rapid development of binding strength in the exposed tar cylinders may be obtained by comparing the stabilities obtained for the tar mixtures with the stabilities obtained on similar cylinders made with slow- and rapid-curing liquid asphaltic materials and exposed at the same time. Cylinders made with 23 slow-curing liquid asphaltic materials that had initial stabilities of from 25 to 175 pounds, had developed stabilities at the end of 15 weeks of from 300 to 3,075 pounds. Cylinders made with six mediumand rapid-curing liquid asphaltic materials had initial stabilities of from 25 to 275 pounds and developed stabilities ranging from 1,650 to 5,200 pounds at the end of 15 weeks.

At the end of 5 weeks the cylinders containing the TC, TM, and TR tars had developed stabilities ranging from 3,600 pounds to more than 10,000 pounds at the test temperature of $25^{\circ} \mathrm{C}$., which was also the control temperature of the tests on the cylinders containing the asphaltic materials. For these cold-application materials, only cylinders containing tars 8,11 , and 14
had lower stabilities after 5 weeks of exposure than the maximum value obtained on the asphaltic materials for 15 weeks of exposure. At the end of 15 weeks, the cylinders containing the cold grades of tar had developed stabilities ranging from a minimum of 1,975 to a maximum of 8,150 pounds at $60^{\circ} \mathrm{C}$., the minimum stability being many times greater than the maximum developed with the asphaltic materials. These data seem to substantiate the fact that tar products in service harden more rapidly and ultimately produce harder and less plastic road mixtures than do asphaltic materials.

## CONCLUSIONS

The data obtained on these tar products when submitted to laboratory tests and when subjected to exposure conditions justify the following conclusions.

1. The road tars submitted by the five producers were, with few exceptions, well within the limits of the various grades of materials meeting the Federal specifications.
2. The Saybolt-Furol viscosimeter is well adapted to the determination of the consistency of fluid tars.
3. The vapor end point of $300^{\circ} \mathrm{C}$. ( $572^{\circ} \mathrm{F}$.) as required by distillation method A.S. T. M. D) $20-30$ is approximately equivalent to a liquid end point of $360^{\circ} \mathrm{C} .\left(680^{\circ} \mathrm{F}\right.$.) as required by distillation method A. S. T. M. D) 402-34 T. However, the control of intermediate cutting points by the temperature of the liquid residue may result in fractions with widely different boiling points so that the character of the distillates from various materials cannot be accurately determined.
4. The greatest difference in results obtained by the two distillation methods is caused by the different methods of pouring the residues. A. S. T. M. method D 402-34 T results in residues that have undergone a greater loss of volatile matter and are correspondingly harder as measured by the softening point and penetration tests.
5. The residues from distillation generally have good ductility at $25^{\circ} \mathrm{C}$., but this ductility is rapidly lost when the temperature of test is reduced.
6. When thin films of these tars were subjected to the exposure conditions of this investigation it was indicated that:
(a) The ultimate loss on exposure approximates the total loss of volatile matter in the A. S. T. M. D 20-30 distillation test.
(b) The greater hardness of the residues from exposure as compared with the hardness of the distillation residues results from causes other than the mere loss of volatile matter.
(c) The increased hardness of the exposure residues was accompanied by an increase in free carbon content much greater than the amount that was caused by concentration, this being attributed to oxidation, carbonization, or both.
(d) The test data indicate that in general the index of increase of free carbon in the residues from exposure is higher for those tars having initially low free carbon contents.
7. While the initial stability or binding value of the sand mixtures containing the more fluid tars is relatively low, it is roughly proportional to the consistency of the tar used.
8. The bonding strengths of the distillation residues are more nearly proportional to their softening point. values than to their penetrations.
9. The development of stability or bonding streng th in the tar and sand mixtures proceeded rapidly under the exposure conditions of this investigation, producing stability values much higher than could be obtained by mere loss of volatile matter and developing specimens which, when broken, gave no evidence of retaining plastic properties.
10. The amount of volatile matter and the consistency of the residues from the laboratory distillation test, together with the behavior of these materials in thin films and in molded specimens, indicate that road tars are rapid-hardening types of bituminous materials.

## PRESERVE BENCH MARKS FOR FUTURE USE

IN THE extension of its system of control surveys over the United States, the Coast and Geodetic Survey has run many hundreds of lines of levels, of first- or second-order accuracy, which now form a great net over the entire country with the lines spaced, except in some of the more inaccessible regions in the West, at intervals of about 25 miles. Bench marks are spaced along these lines at intervals of from 1 mile or less to a maximum distance of 3 to 5 miles.
These bench marks and their published descriptions and elevations represent a vast outlay for field work, computation, and adjustment. In order that the results of this work may be useful to all engineers throughout the Nation the marks must remain in place undisturbed or be relocated in such a manner as to preserve their usefulness.
In spite of the extreme care exercised by the field parties, in placing bench marks where they are likely to remain undisturbed for a long time, it sometimes becomes necessary to destroy them to make way for construction, repair, or maintenance work of various kinds. A bench mark once disturbed, without transfering its elevations to some other mark which is set to preserve it, is a total loss and leaves a gap in the line of levels which requires extra running for all engineers who may be called on to do leveling in the locality at any future time.
It is to the interests of all concerned to relocate bench marks which are in the way of construction rather than to allow them to be completely destroyed. The Coast and Geodetic Survey does not have funds available for placing parties in the field to go about and relocate bench marks which must be moved to avoid their destruction. On the other hand, since the marks are chiefly useful to practicing engineers, whether public or private, we look to all engineers throughout the country for cooperation in preserving these useful marks in their own interests as well as in the interests of their government.
A routine method of handling cases of this sort has been worked out and if followed out, as outlined below, the loss of bench marks and its accompanying inconvenience and loss to the engineers and surveyors of this country will, to a large extent, be avoided.
As soon as it becomes known that a mark must be moved, a letter should be sent to the Director, United

States Coast and Geodetic Survey, Washington, D. C., attention Section of Leveling, stating the necessity for moving the mark and giving its designation. The designation consists of the letters and numbers found to have been stamped with dies on the disk. It is desirable to furnish a rubbing of the disk as well. 1 rubbing can be made by placing a piece of mediumweight paper over the disk and then rubbing over the paper with a hard pencil to bring out the legend cast in the disk, especially the letters and numbers stamped on it with dies.

Upon receipt of this information, this office will send out a new disk properly stamped to show that it has been reset. Necessary instructions for the establishment of the new mark and the transfer of elevation will also be sent. The proper procedure, in most cases, is to establish the new mark in a safe place nearby and transfer the elevation from the old mark to the new one by means of an engineer's level and rod. The levels should be run in duplicate to avoid the possibility of large errors, and all readings should be made to three decimal places in order to preserve the accuracy of the original elevation.

The old mark should not be disturbed until the observations involved in the transfer have been checked by the observer or the recorder. An assumed elevation for the old mark may be used in the transfer, since what we are primarily concerned with in a case of this sort is the difference in elevation between the old mark and the new one established to replace it.

After the new mark has been established and the elevation transferred to it, the old disk should be broken out and returned to this office in a franked mailing sack which will be supplied for the purpose. A complete report on the action taken, including a description of the location in which the new mark is established and a copy of the field notes involved in the transfer of elevation, should also be forwarded to this office; a franked envelope will be furnished for this purpose.

The cooperation which individuals and organizations may extend to this office in preserving the benchmarks will be a service not only to this bureau and other government surveying organizations but to anyone who may have occasion to use the marks.

CURRENT STATUS OF UNITED STATES WORKS PROGRAM HIGHWAY PROJECTS
（AS PROVIDED BY THE EMERGENCY RELIEF APPROPRIATION ACT OF 1935）

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CURRENT STATUS OF UNITED STATES WORKS PROGRAM GRADE CROSSING PROJECTS





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[^0]:    ${ }^{1}$ Prepared Tars and Their Application to Road Construction. Roads and Road Construction, vol, XIII, no. 147. March 1935.

[^1]:    1 The material shall be homogeneous.
    ? Specific gravity of total distillate $38^{\circ} / 38^{\circ} \mathrm{C}$. minimum (water free), 0.96 .
    ${ }_{3}{ }^{2}$ The Federal specifications designate this material as refined hot-application tar. For convenience, the designation TH-1 is used in this report.

    - The material shall be homogeneous and free from water.

[^2]:    No information furnished. Classiflcation based on test results.
    2 Classification based partly on information furnished by producer and partly on test results.

[^3]:    ${ }^{3}$ In the following discussion the TC, TM, and TR grades are referred to as tars for cold application or cold tars, and the TH and TP grades as tars for hot application or hot tars.

[^4]:    ${ }^{1}$ A Discussion of Road Tar Consistency Relationships. Engineering NewsRecord, vol. 111, no. 16, October 19, 1933.

[^5]:    6 Rationalization and Simplification of Test Renuirements for Lis uid Asphaltic
    Materials, by E. F. Kelley and Prevost Hubbard. Public Rostis, vol, 13, no. 6, Materials, hy E. F. Kelley and Prevost Hubbard. Public Roars, vol, 13, no. 6, August 1932.

[^6]:    7 Ductility at Low Temperatures. 1935 Proceedings of the Association of Asphalt Paving Technologists.

[^7]:    The Effect of Exposure on Bitumens, by Prevost Hubbard and C. S. Reeve Industrial and Engineering Chemistry, vol. 5, no. 1, January 1913.

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